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The Journal *of the* Society of Dyers and Colourists

Volume 77 Number 3

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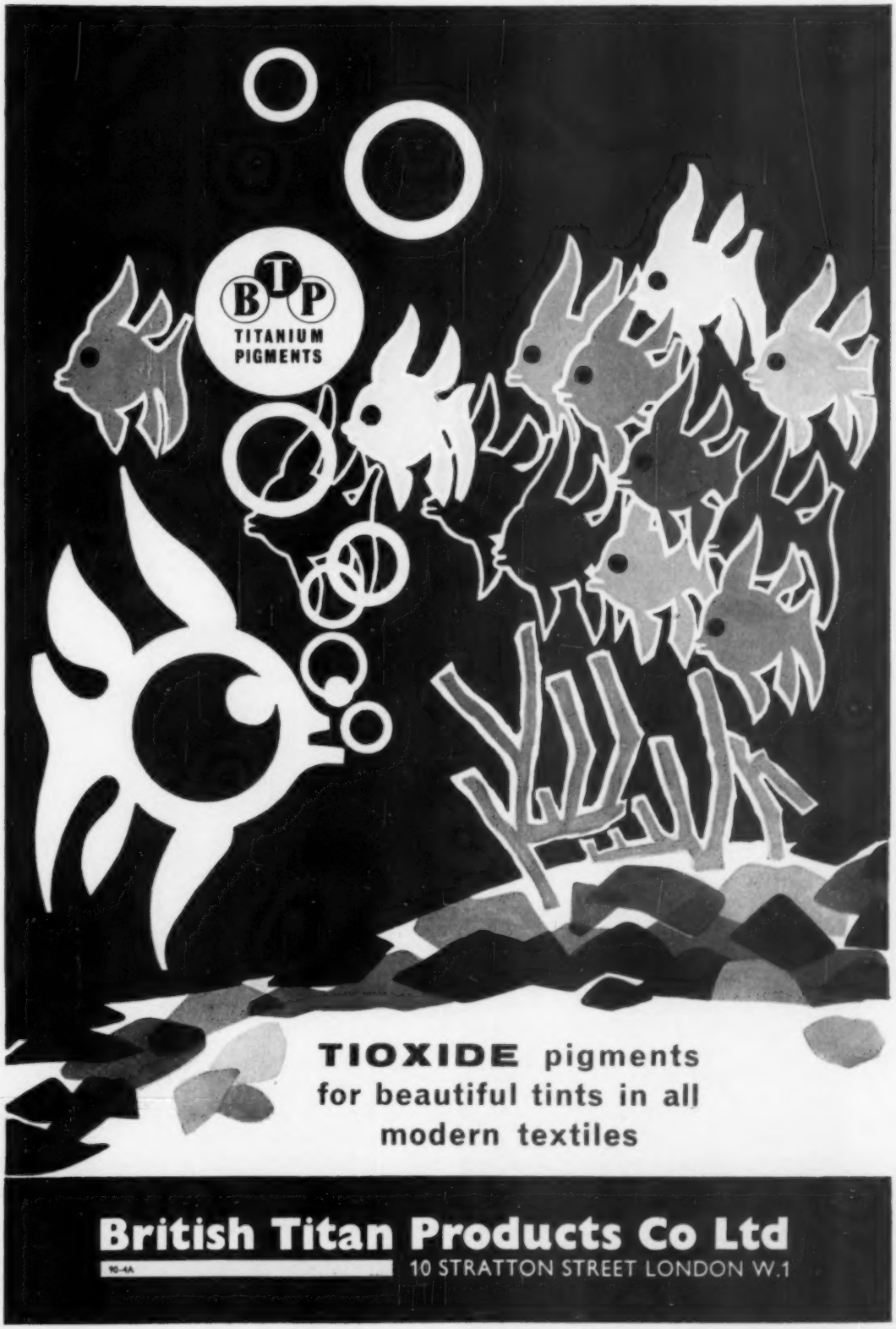
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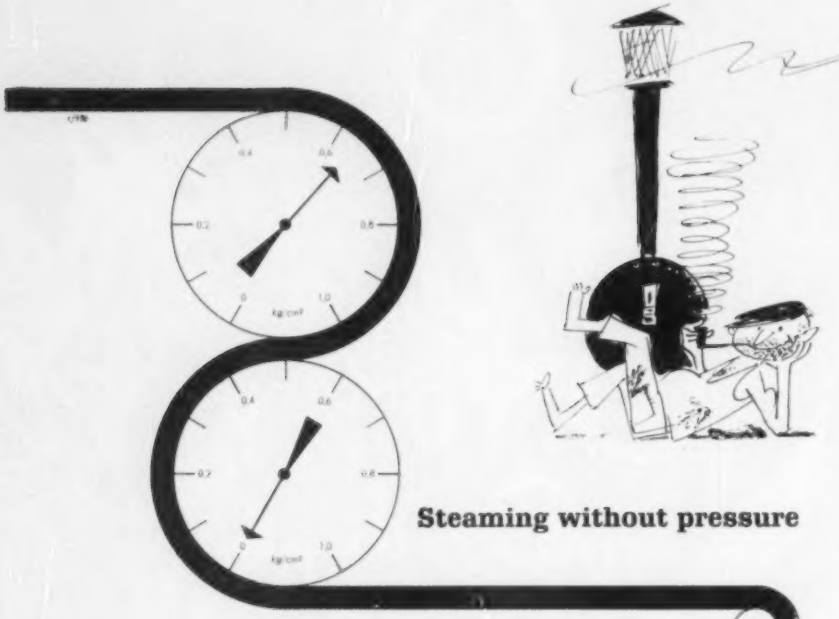

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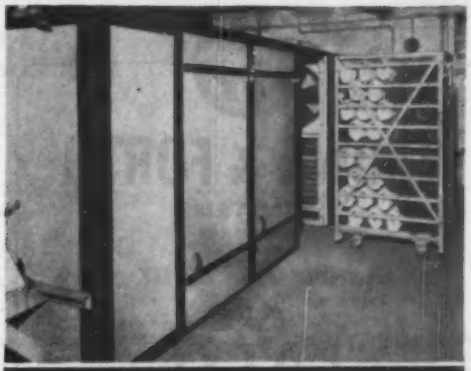

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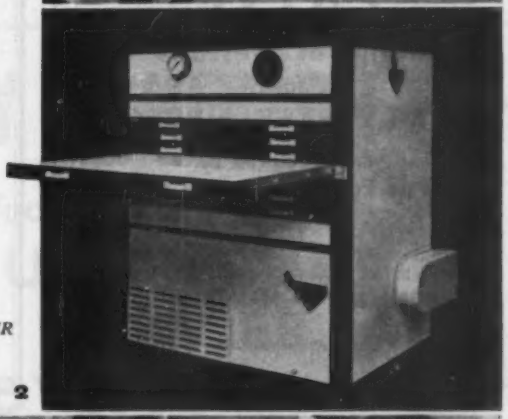

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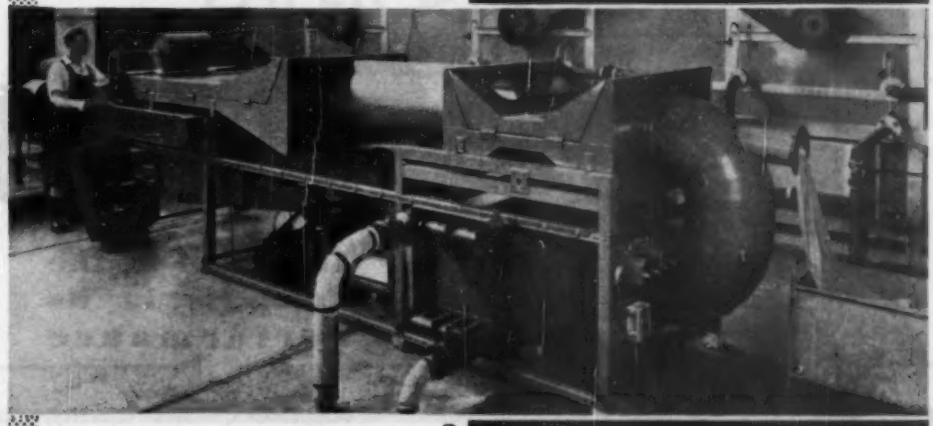

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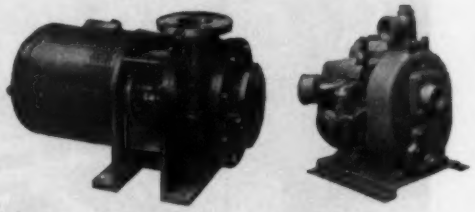

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(Abstracts section only—see January issue, page 2)

NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc, should consult pages 1–8 of the January 1961 and pages 389–396 of the July 1960 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

COMMUNICATIONS

Stripping of Procion Dyes from Cotton for Testing Purposes — *T. P. Nevell*

Setting of Wool Fibres in Dyeing — *R. V. Peryman and R. F. Pickup*

Influence of Polar Groups on the Hydrophile–Lipophile Balance of Non-ionic Compounds — *N. Schönfeldt and U. Almroth*

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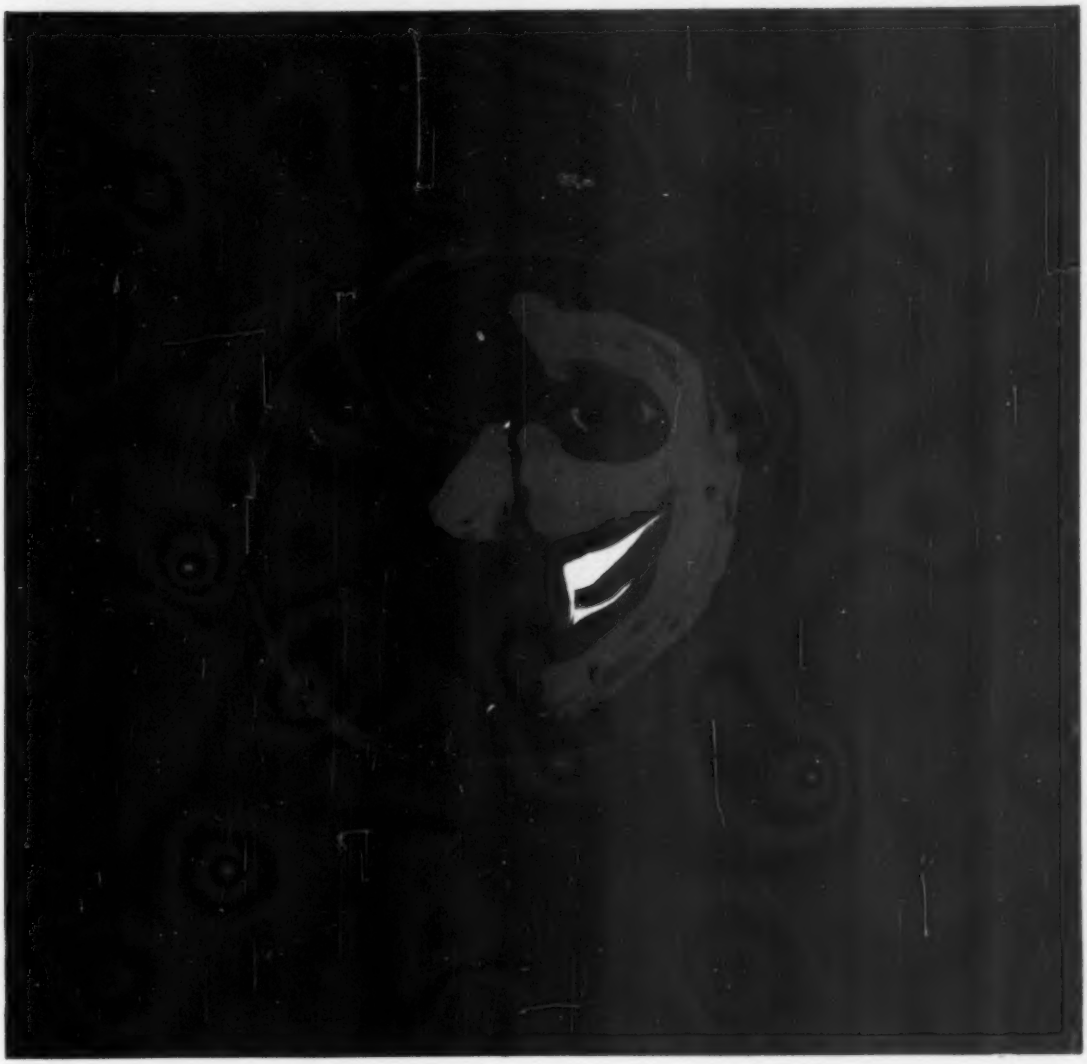

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THE JOURNAL

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Volume 77 Number 3 MARCH 1961 *Issued Monthly*

Proceedings of the Society

Dyeing and Printing Polyester and Polyester–Cellulosic Fabrics with Polyestren Dyes

H. MUSSHOFF

Meeting of the Manchester Section held at the Textile Institute on 20th November 1959
Mr. H. A. Turner in the chair

Polyester and polyester–cellulosic fabrics can be dyed and printed with a range of vat dyes specially selected and processed for the purpose. The application and properties of these Polyestren dyes are discussed, and compared with those of other dyes suitable for dyeing these fabrics.

Introduction

Although at present only small amounts of 100% polyester fabrics are dyed, blends of polyesters with cotton, linen, and viscose rayon are gaining in popularity. The good crease-recovery properties of polyester fibres are shown to advantage in mixtures containing 67% polyester and 33% cotton; fabrics of this composition are used to a considerable extent in shirts, blouses, dressgoods, raincoats, tropical wear, and even military uniforms. Fabrics with polyester warp and cotton weft do not possess the advantages of fabrics made from spun-fibre mixtures. Polyester fibres and linen are usually blended in equal amounts. 100% Polyester fabrics used for blouses and dressgoods are generally printed; polyester marquisettes and knitwear are dyed in pale shades.

Dyeings on these materials must have good fastness to light, water, washing, perspiration, and rubbing, properties which can generally be obtained with vat dyes. It was therefore of interest to see if vat dyes would dye polyesters, the following factors being important—

(*a*) The method of application should be simple, and, if possible, continuous.

(*b*) Carriers should not be necessary.

(*c*) The fastness properties on polyester and on cellulosic fibres should be identical.

The first trials were carried out on the jig using dispersions of acid leuco compounds (triethanolamine + hydrosulphite), as with disperse dyes; because of the poor affinity, only pale shades were obtained, but the depth could be increased by heat-setting. The behaviour of highly dispersed vat dyes, padded on the fabrics as pigments and then heat-set, was next investigated. As the results were very promising, the Indanthren dyes were tested, the most suitable ones being developed as the Polyestren* dyes.

Although there are 160 Indanthren dyes, the Polyestren range at present comprises only nine dyes. This is because a suitable dye, in addition to satisfying the conditions listed above, must—

(*a*) have the same affinity for polyester and cellulosic fibres (or a greater affinity for polyesters)

(*b*) give the same colour on each type of fibre

(*c*) be compatible with all other dyes of the range, and

(*d*) be satisfactorily fixed by heat-setting.

It seems likely that the range will eventually be extended.

Pretreatment of Fabric

Pretreatment of fabric is important. Willinger[1] recommends that fabrics be scoured for 30–60 min at 70°C in dilute alkali and an anionic detergent. If, however, the polyester fibre has been treated with a cationic antistatic agent during processing, any residual agent will form a precipitate which is hard to remove and which leads to the formation of spots and specks during dyeing. If such agents are likely to be present, the fabric must be scoured with non-ionic detergents under acid conditions.

Open-width scouring obviates the need for presetting; if scouring has to be done on the winch, presetting is necessary to prevent rope-marking.

* Registered trade mark of Cassella Farbwerke Mainkur A.G.

Presetting at 210–220°C increases the dye-affinity of the fibre and hence the colour yield, especially with disperse dyes, but uneven presetting leads to unlevel dyeings. Furthermore, residues of size or other materials may be permanently fixed on the fibre during presetting.

It may also be necessary to bleach the fabric, especially for pale and brilliant shades. Generally, the best results are obtained if sodium chlorite is used as bleaching agent on 100% polyester fabrics; other agents may be used with advantage on mixture fabrics.

Polyester–cotton fabrics are often mercerised. Resistance to degradation after mercerisation is good, and the tensile strength is unaltered if the treatment is carried out at 20°C with caustic soda of strength 30°Bé (53°Tw). Polyesters may, however, undergo hydrolysis on treatment with alkalis at the boil, even at concentrations less than those used in mercerising; loss of weight and of tenacity then result[2].

Fabrics to be dyed with Polyestrens may be singed before being scoured and dyed. The dye dissolves evenly within the fabric during heat-setting, and the small pills formed during singeing do not dye more heavily. If disperse dyes are used (exhaustion method) the pills take up more dye and are dyed more heavily.

Dyeing 100% Polyester Fabrics

The following operations are necessary when polyester fabrics are dyed with Polyestren dyes—padding, drying, heat-setting, reduction clearing, and finishing.

PADDING

The fabrics are padded cold with the dye dispersion on a conventional two-bowl machine. Padding is more difficult than on cellulosic fibres, because polyester fibres absorb less liquor; staple fibre is easier to pad than continuous filament. To overcome the difficulties a thickening agent is added. The colour yield depends on the type of agent used and the quantity added. The most suitable agents are starch ethers, alginates, and Solidokoll K (CFM), a synthetic product which is preferable because it does not cause as great an increase in the liquor viscosity as do other thickeners, and does not adhere to the padding bowls and guiding rollers. The most suitable concentration range is 20–30 g/l.; higher concentrations may reduce the colour yield by retarding dye absorption.

Addition of thickening agent to the padding liquor containing highly dispersed dyes causes the dye to aggregate, which would produce a skittery dyeing. Aggregation is prevented by adding 3–5 ml/l. of a dispersing agent of the ethylene oxide type, e.g. Diazopon A, Dispersogen AZ or Dispersol A.

The padding liquor is prepared by adding to the water the dispersing agent, then the dispersed dye, and finally the thickening agent. The thickener must be dissolved and the solution filtered before being added to the padding liquor, in order to prevent formation of lumps. Solidokoll K, which is a liquid, is added as a 10% solution.

Padding takes place at 30–40°C. The pick-up depends on the type of fabric and the type of padding machine; 100% polyester fabric may pick up 35–60% of padding liquor. Padded cloth must be transferred to the drying machine without touching any stationary part of the machines, since stationary rods or bars act like doctor blades and scrape off the padding liquor.

DRYING

A hot-flue or a pin stenter may be used to dry the padded goods[3]; clip stenters produce clip marks on the cloth. A low-speed cylinder drying machine with an infrared heater between the padder and the cylinder has been used for bulk production, and bulk trials have been carried out on a high-speed cylinder dryer running at 120 yd/min; both dryers produced excellent results. Jet-drying machines also are suitable.

It is difficult to say whether a particular type of dryer is suitable, because this depends on dyehouse conditions as well as on the type of machine. The heating system, ventilation, uniformity of heat supply, and distribution of air flow all affect the result. A certain type of dryer may give excellent results in one dyehouse but poor results in another. In general, any drying machine which gives level-dried fabrics when pigment resin dyes or Phtalogen dyes are used, can be used to dry fabrics padded with Polyestren dyes.

To prevent dye migration it is advisable not to use a drying temperature above 60–70°C; the most important factor, however, is even flow of air over the cloth on both sides.

It is important to realise that any unevenness produced during padding or drying can be remedied by washing off the dye. After heat-setting this is impossible; the dye cannot even be stripped off.

HEAT-SETTING

Heat-setting is the most important operation of the dyeing process. During heat-setting the dye dissolves within the thermoplastic fibre and can no longer be removed. The contention that the dye dissolves as well as migrates into the fibre is supported by the fact that the hue of a vat dye on polyester fibre is the same as its colour in organic solvents.

The temperature of heat-setting has an important effect on the colour yield. The optimum temperature range is 210–220°C (approx. 205–210°C on the surface of the cloth). At lower temperatures the colour yield is poor, whilst at higher temperatures the quality of the fabric may be impaired—polyester fibres melt over a narrow temperature range at approx. 250°C. Heat-setting takes 40–50 seconds; a shorter treatment results in a poor colour yield. Moreover, it is unsatisfactory to heat-set for, e.g., two periods of 20 seconds, since time is required for the fibre to become thermoplastic and for dye to migrate into it. If this process is interrupted, setting for a further 40 seconds at least is necessary to improve the colour yield.

The colour yield may also be increased by heat-setting at 180–190°C if urea or Glycein A is added to the padding solution. Unfortunately, this increases the dyeing costs because considerable quantities are required.

Polyestren Scarlet R, Pink B, and Violet BB may be fixed by heat-setting at 180–190°C if they are used singly or with one another. When they are used as shading components with other Polyestren dyes, heat-setting must be done at 210–220°C. At higher temperatures the dyes tend to sublime.

Highly dispersed vat pigments require a higher setting temperature than do disperse dyes, which generally migrate quicker and are more soluble; on the other hand, the disperse dyes tend to sublime strongly during heat-setting, whereas vat dyes rarely do.

Polyester fabrics must, in any case, be heat-set, so it is advantageous to be able to dye and heat-set simultaneously; suitable machines are available in all dyehouses where polyester fabrics are handled [4]. Heat-setting at the temperature required for Thermosol treatment of Polyestren dyes decreases the pilling tendency of polyester staple. Pilling is more severe on fabrics heat-set at a lower temperature.

REDUCTION CLEARING

When 100% polyester fabrics are dyed to pale depths, a reduction bath is unnecessary unless a very bright shade is required. For medium and heavy shades, however, a reduction bath has to be used to remove surplus unfixed dye. The cloth is treated for 20 min at 50°C in a bath containing—

3–5 ml/l. caustic soda 38°Bé (70°Tw)
2–3 g/l. sodium hydrosulphite (sodium dithionite)
1 ml/l. Albigen A

This treatment can be carried out on a jig or winch; in continuous dyeing the first and second boxes of the open-width washers serve this purpose. Vat dye adhering to the fibre surface is reduced and removed, whereas the dye in the fibre is unaffected. The product of this treatment is a dyeing of excellent rubbing fastness.

FINISHING

Finally, the fabrics are rinsed well to remove residues of caustic soda and hydrosulphite. An oxidation bath is unnecessary since no reduction of the fixed dye has occurred. Soaping at the boil is essential, as it increases the brilliancy of the dyeing and removes the last unfixed dye particles. Rinsing follows as the final treatment.

Application of Polyestren Dyes to Polyester–Cellulosic Fibre Fabrics

Polyester–cellulosic fibre fabrics are dyed in a very similar way to polyester fabrics, except that the dye absorbed by the cellulose during padding is fixed in the reduction bath, not during heat-setting. Polyester–cotton (67/33) and polyester–linen (50/50) are more easily padded than 100% polyester fabrics, because the cellulose absorbs more padding liquor. A thickening agent is still necessary, but the quantity required is less. If Solidokoll K is used, a concentration of 20 g/l. is sufficient. Solidokoll K prevents the pick-up of padding liquor by cellulose from being too high, and also prevents dye migrating from polyester to cellulose during drying. Either a two-bowl or a three-bowl padder is suitable. The padding temperature is 30–40°C and the pick-up obtained is 50–80%, according to the type of fabric.

Dyeing and heat-setting are carried out as already described. Investigation has shown that cellulosic fibres are not adversely affected by exposure to a temperature of 210°C for 50–60 s.

Gas-heated steel cylinders have been successfully used for heat-setting. Since contact heating facilitates speedy heat transfer from the cylinder to the cloth, it is not necessary to heat the cylinders above 210°C. On the contrary, a higher temperature may cause the polyester fibres to soften and become sticky. More than one cylinder must be used in order to prevent a "two-sided" dyeing.

When mixture fabrics are dyed, the reduction bath serves not only to free the polyester fibres from surplus dye, but also to fix the dye on the cellulosic fibres. The amount of chemicals required is higher than when 100% polyester fabrics are being dyed. Glauber's salt or common salt is added to prevent bleeding of dye from cellulose into the bath; Albigen A is not added. The quantities used are—

10–22 ml/l. caustic soda 38°Bé (70°Tw)
4–12 g/l. sodium hydrosulphite
6–20 g/l. salt

The temperature is 60–70°C and the treatment takes 30–60 min. The only exception is the dye Polyestren Yellow GG, which needs less caustic soda and is applied at a lower temperature, namely 25°C.

Polyestren dyes can be divided into two groups according to their behaviour on mixture fabrics—

GROUP I
Both types of fibre dyed same depth and hue
Polyestren Yellow GG
Blue BG
Green GG
Brown GR
Grey G

GROUP II
Dyes with higher affinity for polyester fibre
Polyestren Scarlet R
Pink B
Violet BB
Turquoise G

When dyes of Group II are used, it is necessary to dye the cellulose in the reduction bath on the jig or winch to obtain tone-in-tone effects. This can be done by adding dye to the reduction bath or to the padding liquor. If dye is added to the reduction bath, there is a practically unlimited choice of vat dyes, since polyester fibres do not absorb dye from the vat. If, however, dye is added to the padding liquor, careful selection must be made according to the behaviour of the dye during heat-setting. The Indanthren dyes of practical importance to the dyer can be classified as follows—

CLASS I—**No staining or only slight staining of polyester fibres**

(a) Same hue on polyester and cellulose

INDANTHREN	C.I. VAT
Yellow 3GFN	Yellow 12
Yellow 3R	Orange 11
Yellow 3RT	Orange 11
Golden Orange 3G	Orange 15
Scarlet R	Red 29
Brilliant Scarlet E3G	—
Brilliant Scarlet EFR	—
Rubine GR	Red 21
Blue ER	—
Blue 3GF	Blue 28
Olive GG	Green 14
Olive Green EBG	—
Khaki GG	Green 8
Red Brown GR	Brown 8
Maroon BR	Brown 41
Brown GR	Brown 44

(b) Slight difference in hue on polyester and cellulose

INDANTHREN	C.I. VAT	HUE ON POLYESTER
Red GG	Red 23	Yellower
Olive R	Black 27	Redder
Olive 3G	Green 17	Redder
Brown BR	Brown 1	Redder
Grey EB	—	Browner

CLASS II—**Noticeable staining on polyester fibres**

(a) Same hue on polyester and cellulose

INDANTHREN	C.I. VAT
Yellow 4GF	Yellow 20
Yellow F2GC	—
Yellow GGFS	Yellow 10
Blue CLB	Blue 30
Brilliant Green GG	Green 2
Brilliant Green 3GF	—
Brilliant Green 4G	—
Green 4G	Green 12
Brown FFR	Brown 3
Red Brown G	Brown 39
Red Brown RR	Brown 45
Copper HR	—
Brown NGS	Brown 9
Olive T	Black 25
Grey HBR	Black 32
Direct Black RBS	Black 9 (only greys)

(b) Slight difference in hue on polyester and cellulose

INDANTHREN	C.I. VAT	HUE ON POLYESTER
Brilliant Orange GR	Orange 7	Much yellower
Red F3B	Red 31	Yellower
Rubine RS	Red 13	Yellower
Blue RS	Blue 4	Greener
Blue BC	Blue 6	Greener
Blue 5G	Blue 13	Greener
Green Blue FFG	Blue 7	Bluer
Blue Green FFB	Blue 7	Bluer
Bronze GC	Brown 19	Redder
Red Brown R	Brown 31	Yellower
Copper HGR	Orange 8	Yellower
Brown LG	Brown 46	Redder
Black Brown NTS	Brown 17	Redder
Brown 3GT	Brown 26	Redder
Grey CL	Black 31	Redder
Grey EGR	—	Greener

Indanthren Navy Blue BF (C.I. Vat Blue 19) stains polyester heavily, but gives nearly the same shade as on cellulose. The dye is therefore suitable for shading full blues and navy blues.

After being treated in the reduction bath, the fabrics are finished in the same way as vat-dyed cotton cloth, i.e. rinsed, oxidised, rinsed, soaped at the boil, rinsed, and dried.

POLYESTER–SPUN RAYON FABRICS

These fabrics are not yet manufactured to a great extent, but may gain popularity, possibly in ladies' dressgoods or raincoats. It was therefore considered of importance to investigate their dyeing with Polyestren dyes. Because of its high affinity, spun rayon dyes more heavily than cotton, linen, or polyester fibre. Restraining agents were added to the reduction bath to try to obtain the same depth on both types of fibre, but the results were unsatisfactory. Very good results were obtained, however, by increasing the concentration of Solidokoll A in the padding solution from 20 to 30 g/l., in addition to using a restraining agent. This cannot be taken as standard practice, since the dye-affinity of viscose rayon varies with the degree of stretching applied during spinning, and the dyeing formulae have to be adapted accordingly.

CONTINUOUS DYEING OF MIXTURE FABRICS

It has already been noted that one of the main criteria used in the selection of Indanthren dyes for dyeing polyester fabric was their suitability for continuous application as well as for pad–jig dyeing. Until recently, the yardage per shade was not sufficient to justify the continuous dyeing of polyester–cotton fabrics, but recently fabrics for military as well as other purposes have been required in large enough quantities to warrant the adoption of this method, which has proved satisfactory.

The method of application is almost the same as in the dyeing of cellulosic fabrics, except that an additional heat-setting process, which may be considered as a prolonged drying, is necessary. The fabrics are padded with the dye dispersion, dried and heat-set, padded with caustic soda and sodium

hydrosulphite, steamed for 30–40 s at 102°C, and finished on the open-width washer.

If an Indanthren dye is needed to colour the cellulose, it must be added to the padding liquor. It cannot, as in pad–jig dyeing, be added to the reduction liquor. The selection of dyes which cover cellulosic fibres without affecting polyester fibres has already been discussed.

The reduction bath necessary to remove unfixed dye from the polyester fibre follows in the first box of the open-width washer. The amount of dye which bleeds from the cellulose into the reduction bath must be reduced to a minimum. It is therefore preferable to use dyes with a high rate of strike and to add salt to the reduction bath. The quantities of caustic soda and sodium hydrosulphite can be kept low, since the sole purpose of the reduction bath is to clean the polyester fibre and not to reduce the leuco vat dye on the cellulosic fibre. Rinsing, oxidising, rinsing, and soaping at the boil on the open-width washer conclude the process.

Experience has shown that dyeings with Polyestren dyes on mixture fabrics have a very solid appearance, an effect not as easily obtained with other groups of dyes.

TWO-COLOUR EFFECTS ON POLYESTER–CELLULOSIC FIBRE FABRICS

For many years taffetas and poplins have been produced with warp and weft dyed before weaving in different colours. This iridescent effect can be produced on polyester–cellulosic fibre fabrics by padding the cloth with a dispersion containing Polyestren dyes which dye polyesters preferentially and Indanthren dyes which do not dye polyester fibres. The method of application (pad–jig or pad–steam) is exactly the same as described for solid shades.

The Polyestren dyes Scarlet R, Pink B, Violet BB, and Turquoise G, individually or in mixtures, are suitable for dyeing the polyester fibres, whilst Indanthren dyes of Classes I and II are used for dyeing the cellulose. It is also possible to apply Polyestren dyes only at the beginning, and to dye the cellulose in the reduction bath; in this case all the Indanthren dyes can be used, since they do not dye polyesters when employed in the reduction bath.

Immedial Black B Hydrosol (CFM) is suitable for dyeing the cellulose of mixture fabrics black without staining the polyester fibres during heat-setting. The padding and reducing liquors must contain different chemicals from those used with vat dyes. Solidokoll K is not compatible with sodium sulphide, so that sodium alginate is used as the thickening agent in padding. In the reduction bath sodium hydrosulphite and caustic soda are replaced by soda ash and sodium sulphide or sodium hydrogen sulphide.

Three typical formulae are as follows—

(1) 30 g/l. Polyestren Turquoise G paste
8 g/l. Indanthren Brilliant Orange GR supra pdr.
3 ml/l. Diazopon A (BASF)
20 ml/l. Solidokoll K (CFM)

padded cold, dried, heat-set for 40 s at 220°C, given 8 ends at 60°C in reduction bath in the jig with

30 ml/l. caustic soda 38°Bé (1-N special)
6 g/l. hydrosulphite

rinsed, oxidised with bichromate–acetic acid at 60°C, rinsed, soaped at the boil, rinsed.

Padding liquor was not added to the reduction bath. In spite of this, no tailing or listing could be observed during many bulk trials, and countless runs during routine work. The reason seems to be that not all the Indanthren dye padded on to the polyester is fixed during heat-setting; the surplus dye bleeds off in the reduction bath and an equilibrium is very quickly reached.

(2) 16 g/l. Polyestren Violet BB paste
3 ml/l. Diazopon A (BASF)
20 ml/l. Solidokoll K (CFM)

padded cold, dried, heat-set for 40 s at 200°C, given 8 ends at 50°C in reduction bath in the jig with

1·5% Indanthren Yellow 3R pdr. f.f. dyeing (IW)
15 ml/l. caustic soda 38°Bé
5 g/l. hydrosulphite
10 g/l. Glauber's salt

rinsed, oxidised with bichromate–acetic acid, rinsed, soaped at the boil, rinsed.

This process is mentioned for the sake of completeness, but it is not much practised. The reason is that it is difficult to obtain correct matchings, since polyester fibres are stained in the reduction bath containing the vat dye; staining is very slight but sufficient to affect the hue on the polyester. When Polyestren and Indanthren dyes are padded together, a slight staining of polyester fibres takes place, but since the colour has been previously matched in the laboratory, the change in hue has already been considered, and the correct matching is easy to reproduce. If, on the other hand, the polyester fibres are dyed to the correct colour first and the cotton fibres are then dyed, the polyester fibres may be off shade.

(3) 80 g/l. Polyestren Green GG paste
30 g/l. Polyestren Turquoise G paste
80 g/l. Immedial Black B Hydrosol
5 ml/l. Diazopon A (BASF)
2 g/l. sodium alginate (dissolved in 10 parts of water)

padded cold, dried, heat-set for 40 s at 220°C, given 8 ends at 90°C in reduction bath in the jig with

15 ml/l. Sulfhydrate F
6 g/l. soda ash
1 g/l. sequestering agent

rinsed, oxidised with bichromate–acetic acid, no soaping required.

Sulfhydrate F, sodium hydrogen sulphide (NaHS) liquid, has a reduction power equal to that of sodium sulphide crystals and half that of sodium sulphide conc. Sodium hydrogen sulphide is also available in solid form, e.g. flakes, granules, the reducing power being 3–4 times that of Sulfhydrate F. It is also possible to use sodium sulphide, but a softer fabric handle is obtained by using hydrosulphide.

Two-colour effects have been produced on fabrics with warp and weft of 67/33 polyester–cellulosic fibre blended yarn, as well as on fabrics with mercerised cotton twisted warp and polyester filament weft. There is less danger of fabric

irregularities being noticeable after dyeing on the latter type of fabric. If blends are not very carefully and evenly spun, the appearance of two-colour effects is not very attractive. Since barriness of polyester filament is well covered by Polyestren dyes, and cotton also dyes level, the evenness of two-colour effects on this cloth is better.

Printing with Polyestren Dyes

POLYESTER FABRICS

Since Polyestren dyes are fixed by heat-setting, printing is very easy. The Polyestren Printing dyes, which are finely dispersed and specially processed pastes (different from the dyeing brands) are stirred into a stock thickening paste containing a suitable thickening agent, printing oil, and, if necessary, a defoamer. Suitable thickening agents are crystal gum, British Gum, gum tragacanth, alginate, cellulose ethers (e.g. Tylose DKL), and starch ethers (e.g. Solvitose KV).

Very sharp outlines are obtained by using crystal gum, but it is advisable to add a thickener with less body in order to give a more elastic film and to prevent cracking-off after drying. Solvitose KV gives a very good colour yield. Because of the composition of the printing pastes containing Polyestren dyes, their stability is excellent.

After being printed, the fabrics are dried as usual and heat-set at 205°C (cloth surface temperature) for 45–60 s. It is advisable to heat-set the fabric face down so that cracked-off particles do not adhere to the surface of the fabric, where they might be absorbed by the thermoplastic fibre and appear as specks. The fabrics are next rinsed well to remove thickening agent, and are then treated for 15 min at 70°C in a reduction bath containing

3 ml/l. caustic soda 38°Bé (70°Tw)
2 g/l. hydrosulphite
1 ml/l. Albigen A

Finally, the fabric is rinsed, soaped at the boil, and rinsed again.

POLYESTER–CELLULOSIC FIBRE FABRICS

The Polyestren Printing brands are ideal for direct printing of mixture fabrics. Most of these dyes print both fibres in the same depth and hue; only Orange R, Scarlet R, and Pink B require the addition of a suitable Indanthren dye to the printing paste in order to obtain a fuller and brighter shade on cellulose. This addition does not complicate the printing process. A thickener containing alginate and wheat starch is suitable.

Fixation of dye requires two processes, both of which have the advantage of being continuous. The dye is fixed first on the polyester fibres by heat-setting, and then on the cellulosic fibres in the same way as with Colloresin prints on cellulose. Flash-ageing also is suitable.

After being steamed, the fabrics are rinsed well, oxidised, soaped at the boil, and rinsed again. They are not treated in a reduction bath since the prints would bleed into the white.

The range of Polyestren printing brands is as follows—

POLYESTREN PRINTING

Yellow G paste*	Blue G paste
Orange R paste*	Brown R paste
Scarlet R paste*	Green 5G paste
Pink B paste*	Grey N paste*
Violet B paste	

* These dyes may be fixed by steaming (see section on Discharge Printing below).

To produce a black on mixture fabrics, a dye mixture is used, because no black dye is yet available. The composition is—

15 g Polyestren Printing Yellow G paste
27 g Polyestren Printing Pink B paste
28 g Polyestren Printing Violet B paste
27 g Polyestren Printing Blue G paste
105 g Indanthren Printing Black LF Suprafix paste (BASF)

Discharge Printing

Polyestren Printing dyes are also suitable for discharge printing. Heat-setting is the best procedure for fixing them, but those marked with an asterisk in the list above may be fixed by steaming for 10–20 min at 15–28 lb/in² in a star ager if Decroline soluble conc. (100 g/kg) is added to the printing paste. The colour yield of prints fixed by steaming is lower than that of heat-set prints. This discontinuous printing method will therefore be limited to discharge styles and will rarely be used for direct printing.

It is also possible to produce coloured discharges by the Thermosol process (heat-setting) using Decroline soluble conc. A better colour yield is thereby obtained. All Polyestren Printing dyes are suitable. Two procedures are possible—

(*a*) production of coloured discharges on dyeings with disperse dyes

(*b*) production of coloured discharge resists on paddings with disperse dyes.

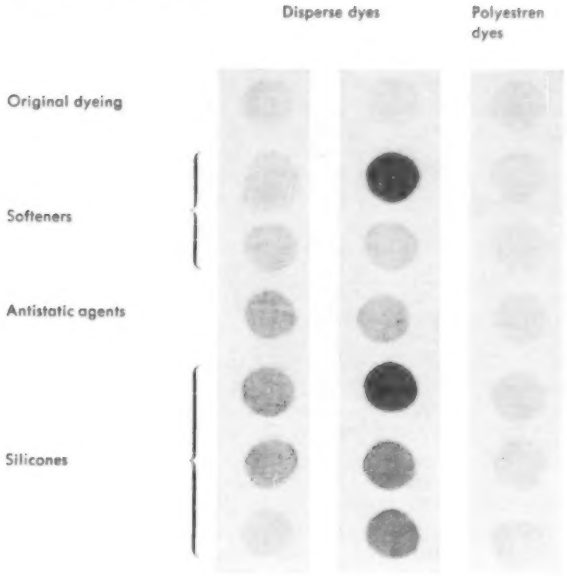
Both methods are relatively easy and may be made continuous.

The coloured discharges contain only dye, thickener, and Decroline sol. conc. Production of certain dyeings with disperse dyes requires the addition of anthraquinone and triethanolamine to reinforce the discharge. A mixture of crystal gum and a starch ether is a suitable thickener. After the discharge has been printed on to the fabric, the cloth is dried and heat-set on a pin stenter, the disperse dye being destroyed by the discharge and the Polyestren dye fixed.

For discharge resists on fabrics padded with disperse dyes, fixation of both dye groups and the discharge of the disperse dye take place simultaneously during heat-setting. In contrast to the steaming process, in which it is difficult to prevent haloing, heat-setting produces sharp outlines of the white or coloured discharges. Reoxidation of the prints is unnecessary, since the Polyestren dyes are practically unreduced during the process. A reduction bath to remove surplus dye leads to good rubbing fastness; it is followed by rinsing and soaping.

It is also possible to print disperse dyes along with Polyestren dyes by direct printing on polyester fabrics.

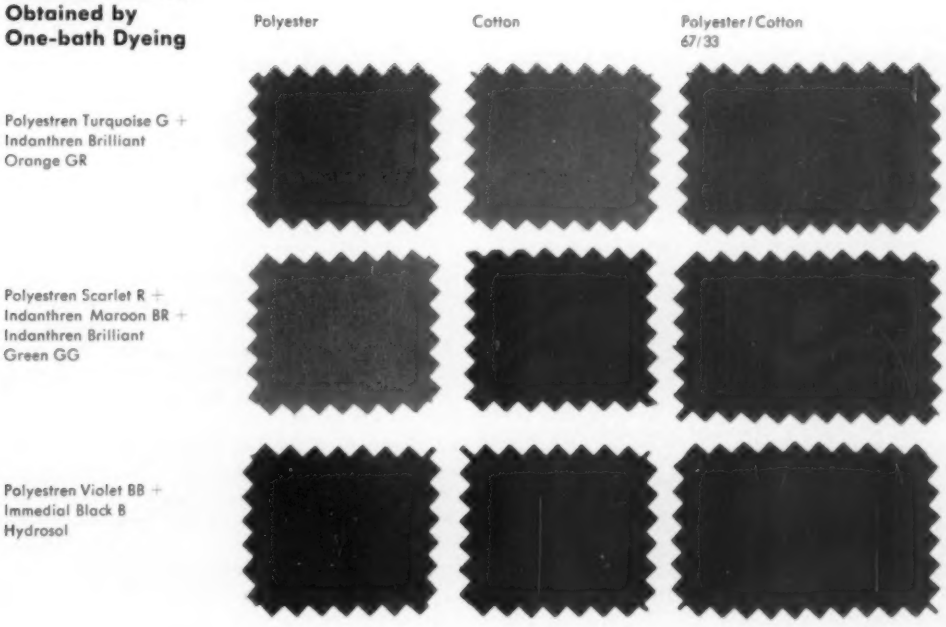
Influence of Finishes on Rubbing Fastness



Iridescent Effect on Fabric of Cotton Warp and Polyester (c-f) Weft Dyed in One Bath

Polyestren Turquoise G + Indanthren Brilliant Scarlet EFR

Two-colour Effects Obtained by One-bath Dyeing



Concluding Remarks

Dyeing and printing are relatively easily performed with Polyestren dyes. Some adaptations have to be made to existing machines, a not unusual procedure when a new fibre is developed. Somewhat more serious are the difficulties originating from the polyester fibre itself. In common with polyamides and viscose rayon, polyester fibres have a dye-affinity which increases with decreasing degree of stretching applied during spinning. A formula developed for dyeing or printing one type of polyester fibre may not give the same result on other types. Furthermore, polyester fibres absorb only a limited amount of dye; once the fibre is saturated with dye, the application of more dye does not lead to heavier shades. This is true not only for Polyestren dyes, but also for disperse dyes. When disperse dyes are applied, carriers and the use of higher temperatures make it possible to obtain heavy shades, but carriers have the disadvantage that they cannot easily be completely removed from the fabric. Carriers may be toxic and their odour may be imparted to the fabrics.

Because of the low dye-affinity of polyester fibres and the tendency not to use carriers, it is impossible to dye very heavy shades on all types of polyester with Polyestren dyes; pale and medium shades and rather heavy blues and browns have been dyed successfully, but so far not a black.

Formulae for dyeing and printing have to be developed in the laboratory on representative samples of the material to be dyed in bulk. This is not unusual, and is done for continuous dyeing methods. If works conditions, e.g. pick-up, heat-setting temperature, are closely imitated in the laboratory, reproduction of the colour in bulk is not difficult.

The fastness properties of Polyestren dyes on polyester fibres are generally very good, viz.—

Agency	Fastness
Light	5–7
Washing—boil	4/5–5
Perspiration	5
Chlorine	4/5–5
Rubbing—dry and wet	4/5–5
Ironing—dry and wet	5
Solvents	4/5–5

Fastness to rubbing may be detrimentally influenced by softening agents, especially silicones, although Polyestren dyes are far less affected than disperse dyes.

When the good fastness properties, as well as the simplicity of application—especially on mixture fabrics—are considered, it is seen that the Polyestren dyes are of considerable interest to the dyer and printer.

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(*MS. received 2nd May 1960*)

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[3] Müller, J., *Melliand Textilber.*, **40**, 773 (1959).
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Discussion

Mr. L. MORRIS: What hardness of bowls is suitable for the padding process?

Mr. MUSSHOFF: Padding bowls of conventional hardness (60–80 shore) can be used. In our experience it is not necessary to have special bowls for padding polyester or polyester–cotton mixture fabrics.

Mr. E. MOLLOY: What is the viscosity of the padding liquor?

Mr. MUSSHOFF: We did not measure the viscosity of the padding liquor exactly. The quantity of thickening agent to be added was determined empirically. Alginate solid (4 g/l.) or Solidokoll K (20 g/l.) gives good results. The viscosity of such padding solutions is rather low.

Mr. MORRIS: Would passage through a hot Schreiner bowl or hot rollers of embossing calenders fix the Polyestren dyes?

Mr. MUSSHOFF: I do not think so. In spite of the temperature which can thus be obtained, the time is too short for the polyester fibre to become thermoplastic and to allow the dye to migrate into the fibre.

Mr. E. H. EDGE: As the standard method of drying on roller-printing machines in this country is by cylinders, is there any danger of difficulties arising, as experienced in the dyeing process?

Mr. MUSSHOFF: We have used cylinders successfully in drying fabrics padded with Polyestren dyes, but in this case an infrared heater was attached to the machine in front of the cylinders. The infrared heater dries the fabric to such an extent that there is no danger of the thickened padding solution sticking to the cylinders.

Dr. P. W. CUNLIFFE: Would there be a possibility of applying the Polyestren dyes to a sheet of yarn, as in ball-warp processing?

Mr. MUSSHOFF: We have no experience yet in applying Polyestren dyes on Terylene filament in this way.

Dr. A. P. LOCKETT: It has been stated that there are no similarities in the structures of the dyes that were useful. Is the saturation value on the fibre the same for all dyes? If the answer is no, is there any relation between the saturation values and the size of the dye molecule?

Mr. MUSSHOFF: The saturation value on the fibre is not the same for all dyes. We have not yet found any relation between molecular size and saturation value; it seems that certain chemical groups in the molecule are responsible for the saturation value of the dye. The higher the solubility of a dye in water, the higher is the saturation value, even if the dye is applied as a pigment. This refers to vat dyes; my comments do not refer to disperse dyes.

Mr. H. BELLIS: (1) What are the prospects of processing slub mixtures? (2) In regard to badly woven fabrics, it is sometimes an advantage to pre-set; is this possible? (3) Does infrared fixation at the same temperature take less time than fixation by hot air?

Mr. MUSSHOFF: (1) The prospects of dyeing slub mixtures are very good. All that is needed is suitable machinery. Recently we have even dyed polyester staple (loose stock) with Polyestren dyes.

(2) It is possible to dye pre-set fabrics with Polyestren dyes. We have done so in Manchester, where the fabrics had to be scoured in rope form and had to be pre-set to prevent rope-marking. The only disadvantage is that the heat-setting of the fabric and the fixing of the dye require two operations instead of one. Working in this way is more costly, and one advantage of the Polyestren dyes is lost, namely, the possibility of heat-setting and fixing the dye in one and the same operation.

(3) Infrared fixation can be quicker than hot-air fixation, since the cloth is more quickly heated to the required temperature. It is difficult to state how much faster infrared fixation is; materials covered with certain dyes reflect infrared radiation more strongly than do others, and therefore heating takes longer.

Mr. H. R. HADFIELD: It is true that, considered as ranges, vat dyes are faster than disperse dyes. However, there are a few selected disperse dyes which, on my tests, are faster than the thioindigoid vat dyes. What is the possibility of using these selected fast dyes to extend the range of vat dyes?

Mr. MUSSHOFF: Fast disperse dyes are suitable to extend the range of vat dyes, especially in printing of 100% polyester fabrics. Both ranges of dyes can be printed together. As it is still not possible to obtain heavy shades on polyester with Polyestren dyes, disperse dyes have to be used for black, navy blue, bottle green, dark brown, etc.

In dyeing mixture fabrics it is more difficult to use disperse dyes next to vats. Pure polyester is dyed with one of the groups. Mixture fabrics can be dyed with disperse dyes (on polyester) and vats (on cotton) by padding, fixing, and reduction (to clean the polyester and to dye the cotton). This process, however, does not lead to such a solid appearance of the dyeings. Further, when disperse dyes are padded together with vat dyes on mixture fabrics, cotton picks up more liquor than polyester, and disperse dye on cotton has to be washed off and is lost. It is more profitable to use Polyestren dyes on mixture fabrics whenever possible, and to use disperse dyes for heavier shades which cannot be obtained with Polyestrens.

Mr. HADFIELD: Can the lecturer comment on the use of vat acids by the Thermosol technique? It is my experience that the vat acids of thioindigoid reds diffuse more rapidly, and yield heavier shades more easily than the actual vat dye. Can this technique be used for the production of heavy shades? It seems illogical to apply fast-to-sublimation dyes in pale shades and then, in heavy shades, where the highest sublimation fastness is required, to use a dye of lower sublimation fastness.

Mr. MUSSHOFF: It is, of course, not logical to use the faster-to-sublimation dyes for pale and medium shades and the less fast ones for heavy shades. Naturally, we would like to use Polyestren dyes for dyeing heavy shades, too, but owing to the lower saturation value (in comparison with disperse dyes) the possibilities are limited.

We have tried to apply dyes as vat acids, but we could not obtain a better colour yield nor could we obtain heavier shades. This, of course, is true only for the comparison of vat acids with the highly dispersed Polyestren dyes. If we compare vat acids with ordinary vat dyes, the vat acids are superior. Polyestrens are more easily applied than vat acids. First, the time and work required for preparing the vat acid are saved; secondly, it is not always easy to re-oxidise a vat acid at the end of the Thermosol process. Some vat acids oxidise readily during drying, others do not; it may be necessary to re-oxidise the vat acid on polyester in a boiling bichromate–acetic acid bath. This is not necessary with Polyestren dyes.

Mr. P. J. DOLBY: It was mentioned that in the dyeing of mixture fabrics it is sometimes necessary to shade the cellulose component with vat dyes when using the Pink and the Scarlet dyes owing to the heavier shade obtained on the polyester component. Is it similarly recommended, when any of the other dyes are used, to shade the polyester component with disperse dyes in medium to heavy shades? Has this technique been used to produce the grey military shades exhibited?

Mr. MUSSHOFF: If the polyester fibre in a mixed fabric is off-shade, it is possible to shade this component with disperse dyes. This technique has not been used to produce the grey military shade exhibited during my lecture; this shade was dyed with Polyestren and vat dyes only. Both dyes were padded together on the mixed fabric, dried, heat-set, treated in a reduction bath (jig) to fix the vat dyes on cotton, oxidised, and soaped at the boil.

New Observations on the Tippy Dyeing of Wool

H. R. HADFIELD and D. R. LEMIN

Meetings of the Scottish Section held at the Royal College of Science and Technology, Glasgow, on 1st November 1960, Mr. J. G. B. McCallum in the chair; of the West Riding Section held at the Victoria Hotel, Bradford, on 10th November 1960, Mr. J. Rankin in the chair; of the Huddersfield Section held at Silvio's Cafe, Huddersfield, on 15th November 1960, Mr. G. H. Binns in the chair; and of the Midlands Section held jointly with the Leicester Branch of the Guild of Technical Dyers at the Colleges of Art and Technology, Leicester, on 16th November 1960, Mr. A. S. Cluley in the chair

New information on the relation between dye constitution and behaviour in dyeing skittery wools is provided. It is shown that the production of skittery dyeings depends upon (*a*) the hydrophilic/hydrophobic properties of the wool surface and (*b*) the hydrophilic/hydrophobic balance of the dye. Information on the hydrophilic nature of the wool surface was obtained by contact angle measurements and on the hydrophobic properties of the dye by measurements of partition between water and butyl alcohol saturated with water.

Introduction

DEFINITION OF THE PROBLEM

Unlevelness in dyed wool may be of one of three types—

(*a*) Unlevel dyeing in which large areas are more, or less, heavily dyed than the bulk of the material. This type of unlevel dyeing is not peculiar to wool and is caused by uneven liquor circulation, too rapid absorption of dye, or uneven temperature or pH distribution during dyeing.

(*b*) Uneven dyeing caused by gross differences in dyeing behaviour between large areas of the material as a result of uneven chemical or physical modification, e.g. uneven carbonising or uneven abrasion of the wool fibre surface.

(*c*) Uneven dyeing resulting from random differences in dyeing behaviour from fibre to fibre or even along a single fibre. This is known as skittery dyeing and one cause of it is the subject of this paper. The other cause of skittery dyeing of wool is the use of a blend of widely different qualities where, even if the fibres were all uniformly dyed, they would appear to be dyed to different depths because of the well-known optical effect on fibres of different thickness. The paper is not concerned with this cause of skittery dyeing.

Skittery dyeing caused by variations in dyeing properties of a single wool fibre along its length is known as "tippiness" or "tippy dyeing". Loose wool is still in essentially the same form as when on the sheep's back, and the tips of the fibre, which have been exposed to weathering and mechanical action during growth, dye differently from the body and root ends of the fibre, which have been protected. This problem has been the subject of much study, and, although considerable evidence has been accumulated and, quite recently, a more satisfactory explanation of skittery dyeing has been offered, this does not provide an interpretation of large amounts of empirical information, well known to the dyer, on the relation between the covering of "tippiness" and the dyes or dyeing methods used.

PRELIMINARY WORK AND INITIAL THEORIES

Tippy dyeing was first discussed by von Bergen [1], who reported both "positive" and "negative" dyeing effects. In the first case the tips were dyed more heavily than the roots and in the second case less heavily. Von Bergen found that milling acid dyes tended to give "positive" tippy dyeing, whilst level-dyeing acid dyes generally gave "negative" effects. Race *et al.*[2] suggested that the different effects resulted from differences in the colloidal properties of the dyes. The highly aggregated, colloidal milling acid dyes were unable to penetrate the undamaged root ends during the initial stages of dyeing, but could penetrate the degraded and swollen tip ends of the wool. Since the milling dyes possess only poor levelling properties, diffusion of dye from the heavily dyed tips to the lightly dyed roots occurred only extremely slowly, and in normal dyeing times, results which were still "positive" were obtained. With the level-dyeing acid dyes, which have low molecular weights, it was suggested that, after initial "positive" distribution resulting from increased adsorption of dye on the accessible tip areas, dye diffused out of the tips during boiling more readily than out of the roots and there finally resulted a "negative" dyeing. Later Vickerstaff[3], whilst agreeing that the explanation of the action of milling acid dyes was feasible, concluded that the explanation of the mode of action of level-dyeing acid dyes was much less satisfactory, since it is difficult to imagine that a purely kinetic effect could alter the equilibrium distribution between tip and root. It must be assumed, therefore, that chemical changes in the tip have given rise to a fibre having lower affinity for dyes than the undamaged root. It is not known whether this explanation is correct, since there does not seem to be any information on the titration curves of separate tip and root wool, nor have dyeings with milling acid dyes been carried to equilibrium.

Evidence is available that, when chrome dyes are used, metal ions are adsorbed to different extents on different areas of the fibre; Race *et al.*[2] showed that the tips took up more metal ions than the roots. Millson[4] used chrome dyes which in the unchromed state were not tippy dyeing, and showed that the addition of small amounts (1–5 p.p.m.) of various metal ions resulted in metal-lake formation in the tips but not in the body of the fibre. Further confirmation was obtained by taking autoradiographs of wool fibres treated with radioactive metal salts, which again revealed the preferential

absorption of radioactive ions in the tip areas of the fibres. Clearly, the distribution of metal ions in the fibre will influence the distribution of chrome dye, and this secondary effect will be superimposed on the primary effect previously outlined.

There can be no doubt, therefore, that "tippiness" is the result of the different adsorption behaviour of the tip and root ends of the fibre. Barr *et al.*[5] isolated heavily and lightly dyed fibres from a skittery dyeing and showed that the heavily dyed fibres were the weaker and also swelled more in aqueous solution. At this time, views on the causes of tippy dyeing appeared to stabilise. It was generally accepted that the tip of the wool fibre was damaged, resulting in greater swelling power, and the swollen tip was thereby made more accessible to dye molecules. Prolonged dyeing, or the effect of metal ions, could complicate matters, but did not affect the primary cause of tippy dyeing.

FURTHER ADVANCES—IMPORTANCE OF FIBRE SURFACE

More recently, in America, Millson and his coworkers[6–9] have studied the problem of skitteriness in great detail using microscopic methods. Millson, Royer, and Wissemann[6] showed that in a skittery dyeing four types of fibre could be detected—undyed, partially dyed, completely but lightly dyed, and completely and heavily dyed. This behaviour was attributed to differences in degradation between the tips and roots of individual fibres, and between fibres from different portions of single fleece, and to the presence in the blend of fibres, differing in diameter and other physical factors, obtained from different sheep. Watkins, Royer, and Millson[9] extended the study. They took samples of dyed wool and determined the percentage of fibres falling into the four groups found by Millson, Royer, and Wissemann. This was done with a number of dyes, samples being taken at different stages during the dyeing process. Level-dyeing acid dyes and milling acid dyes showed marked differences in behaviour. With all the level-dyeing acid dyes, ring-dyed fibres first appeared and increased in number to a maximum with increasing temperature. With further increase of temperature and time, the number of ring-dyed fibres decreased as penetration occurred and this decrease corresponded to the increase in uniformly dyed fibres. A few fibres became heavily dyed at one stage in the process, but these soon lost dye to less heavily dyed fibres, and at the end of the dyeing all the fibres were uniformly dyed. With milling dyes, the behaviour differed markedly from that occurring with level-dyeing acid dyes, and individual dyes showed considerable variations. Evidence was also provided to support the idea that there is a critical temperature for each dye below which little or no dyeing occurs.

Royer *et al.*[8] showed that the addition of undyed wool to uniformly dyed wool produces a much greater reduction in the apparent visual depth of the mixture when blended than would be produced by redistributing the total dye present. This accounts for the loss of depth observed in skittery dyeings. It is well known that if a skittery wool dyeing is boiled in a blank dyebath until complete penetration is achieved, the apparent visual strength of the dyeing increases; this was demonstrated by Royer and Millson[9] and by Goodall[10].

Royer *et al.*[8] suggested that permeability of the outer scale structure was the most important factor leading to variations in dye adsorption. They showed that treatment of wool with alcoholic caustic potash almost completely eliminated the fibre-to-fibre differences which were apparent when similar untreated wool was dyed under the same dyebath conditions. Indeed, the Kiton Red test for chlorinated wool is based on observations of this type, as the chlorination process is thought to effect removal of the outer layers of the fibre and, as a result, make the fibre more accessible to dyes at low temperatures.

In 1948, Lindberg *et al.*[11] isolated a layer now known as the epicuticle from the surface of wool fibres, and in 1949 Lindberg and Gralén[12] suggested that this epicuticle, a thin layer differing in reactions from normal wool keratin, lies over the normal scale structure of the fibre. This layer was said to be responsible for the exceptionally low frictional properties of wool (in the scale direction) since, when the layer was damaged and removed from the fibre, the interfibre friction increased. Although the cuticular layer of wool is known to be hydrophobic and is recognised as the limiting factor in the penetration of large molecules into the fibre, it has been suggested[13] that, owing to its position as the outermost layer overlying the scales proper, the epicuticle probably makes the greatest contribution to the impermeability of the cuticular layer. Difficulties in wetting intact fibres, the difficulty of dye penetration, and the resistance of wool to enzymatic digestion are ascribed by these workers to the presence of the epicuticle. Marked changes in the wettability of wool may be produced by treatments which increase the permeability of the epicuticle or loosen or remove this layer. Again, Lindberg *et al.* suggest that end-staining and tippy dyeing can be overcome by uniformly damaging the epicuticle. Gralén[14] has gone so far as to state that "if the epicuticle were complete probably no dye would penetrate the fibre".

Further evidence for the effect of the surface of wool fibres on dyeing properties has been provided by Millson and Turl[15], who, using a microscopic technique, found that in the initial stages of dyeing, adsorption of dye takes place on the distal edges of the scales. Knots were tied in wool fibres; during the dyeing process the distal edges on the outer periphery of the loops were raised away from the fibre to give a ratchet-like effect, and these areas were the first on to which dye was adsorbed. Likewise, if a normal, undegraded wool fibre was nicked with a sharp blade, dye penetration occurred, initially, only from the cut in the surface of the fibre. More recently, Millson[16], using a Microdyeoscope, produced further evidence in support of the idea that the epicuticle of the fibre is the real barrier to dye penetration. His results showed that areas of atrophy, degradation, or

damage in wool fibres were the regions in which dye was rapidly adsorbed. Removal of the epicuticle by chemical methods resulted in more rapid sorption of dye at low temperatures and in uniform sorption over the length of the fibres.

This more recent work suggests, therefore, that the relation between tippy dyeing and accessibility of different regions of the same fibre or of different fibres is dependent not so much on chemical differences in the body of the fibre as on the presence or absence of the epicuticular layer on the fibre surface. That there is a surface barrier hindering easy access of dye has been demonstrated by Medley and Andrews[17], who derived a mathematical treatment and obtained experimental evidence to support the theory.

Normal wool fibres are strongly hydrophobic, and this clearly has an effect on the adsorption of typical polar dyes. It is well known that when the wool fibre is made non-felting by chlorination under either acid or alkaline conditions, it becomes more hydrophilic and adsorbs polar dyes much more rapidly than does untreated fibre. Preston and Saha[18] demonstrated that chlorination reduced the flotation of wool fibres, and that at pH 10 the flotation became equal to that of casein fibres. They also found that the root ends of wool fibres floated better than the tips, an observation which suggests that at the tips, where the epicuticle is damaged, the fibre is more hydrophilic than at the roots, where the epicuticle will be intact. Kärrholm and Lindberg[19] also showed that treatment of wool with alcoholic potash or chlorine, which is known to remove the epicuticle, changed the fibre surface from weakly polar and hydrophobic to a condition where it was hydrophilic and permeable to polar compounds.

Very recently, Köpke and Nilssen[20] have presented further evidence of the existence of a surface barrier to the diffusion of dyes into wool; they suggest a chemical constitution for the hydrophobic epicuticle. In addition, they make the interesting suggestion, and detail supporting evidence from a microscopic study, that hydrophilic dyes (e.g. Neolan Pink BA; C.I. Acid Red 186) cannot penetrate the hydrophobic wool epicuticle.

QUESTIONS REMAINING UNANSWERED

Thus there is now strong evidence that the epicuticle of the fibre is primarily responsible for the difficulties encountered during the initial stages of dyeing. The most important evidence is based (*a*) on the dyeing behaviour of wool from which the epicuticle has been removed and (*b*) on microscopic studies. In addition, although Köpke and Nilssen[20] have made a start, little attention has yet been paid to the implications of this new theory on dye structure and dyeing techniques.

Answers to the following questions obtained on the theory that tippy dyeing is caused by greater swelling of the wool tips are not satisfactory.

(*a*) Can direct evidence be obtained which links areas of entry of dye into the fibre with the hydrophobic nature of the wool surface?

(*b*) What is the explanation of the improved coverage of tippy-dyeing wool when monosulphonated level-dyeing acid dyes are used instead of di- or tri-sulphonated level-dyeing acid dyes?

(*c*) What is the explanation for the improved coverage of tippy-dyeing wool obtained by dyeing in the presence of certain cationic auxiliary products? These agents form a complex with the dye and, if the theory were correct that greater accessibility of the fibre tip results from greater swelling in the dyebath, an increase in molecular weight of the complex would lead to greater differences in absorption, not smaller, as are found in practice.

(*d*) Why do certain high-molecular-weight milling acid dyes show better coverage of tippy-dyeing wool than low-molecular-weight level-dyeing acid dyes? The same anomaly exists here as in (*c*).

If satisfactory answers could be obtained on the new theory of tippy dyeing, they would provide additional support for the theory.

Experimental and Results

MEASUREMENT OF ANGLE OF CONTACT

It has been indicated that it would be desirable to correlate the hydrophobic properties of the surface of the wool fibre with the areas of initial penetration of dye. One method of obtaining a quantitative measure of the hydrophobic nature of a surface is to determine the angle of contact of the surface with a liquid. The simplest method for a solid in contact with a liquid is the plate method[21], in which a solid plate of the material is tilted in the liquid to an angle at which the surface, at the point of contact of the solid at the air–liquid interface, remains horizontal. Adam[22] had adapted this method to determine the angle of contact of a single textile fibre in his study of wetting and detergency, and for his modification an accuracy of the same order as that of the original is claimed. It was decided to adopt this modification to determine the hydrophobic properties of different areas of the same wool fibre and also to compare wool fibres after chemical treatments, which are thought to change the hydrophobic properties of the fibre surface.

The apparatus used was identical with that described by Adam[22]. Like Adam, we used an oil–water interface, instead of an air–water interface, because this aided microscopic observation of the fibre at the interface. Transformer oil (20 S.A.E.) was used as it is a clear, water-immiscible liquid of low viscosity.

Two samples of wool were used—(*a*) crossbred slubbing (46–48s quality, mean fibre diameter 33·15 μ), (*b*) unscoured loose wool (52s quality, mean fibre diameter 31·25 μ) which yielded very tippy dyeings. These samples were extracted with ether and then with alcohol, and were rinsed in distilled water before being dried and conditioned at 70°F and 65% r.h. Portions of the scoured, tippy-dyeing loose wool were dyed with 1% Coomassie Blue RLS (C.I. Acid Blue 92) and with 1% Procion Brilliant Red 5BS (C.I. Reactive Red 2). The wool was dyed for 15 min at 100°C; Coomassie Blue RLS was applied from a bath containing 1%

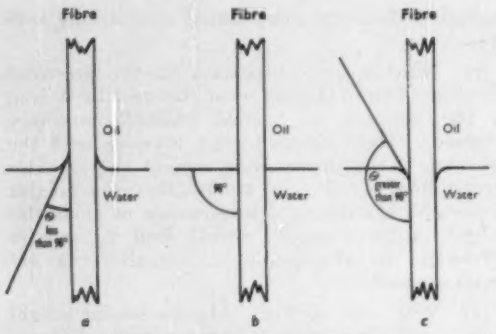


FIG. 1—Diagrammatic representation of fibres at an oil–water interface

glacial acetic acid, and Procion Brilliant Red 5BS from one containing 1% formic acid (85%).

If a fibre is immersed in water, the water will assume a definite angle of contact which depends on the hydrophobicity of the fibre surface. This angle of contact determines the shape of the meniscus immediately adjacent to the fibre; the three possibilities are illustrated in Fig. 1. If air is replaced by a water-immiscible oil, the same pattern of behaviour is observed. A low angle of contact corresponds to a very hydrophilic surface (Fig. 1*a*); a high angle of contact corresponds to a very hydrophobic surface (Fig. 1*c*); and an angle of contact of 90° to the special case where the surface has an equal affinity for the oil used as for water, or is equal in hydrophobic and hydrophilic character (Fig. 1*b*).

Measurements of contact angle were made on the undyed wool slubbing and on both the dyed and undyed areas (the dyed areas being located at the tip) of the tippy-dyeing wool. Dyes were chosen which showed very marked tippy-dyeing behaviour when no auxiliary products were added to the dyebath. The results obtained are detailed in Table I.

In these experiments, the fibre was entered into the liquid vertically; with normal slubbing and the undyed areas of the tippy-dyeing loose wool, a definite "blip" occurred where the fibre passed through the oil–water interface. On rotation of the fibre this "blip" altered in form until a position was reached at which the surface became perfectly flat right up to one side of the fibre. This position was well defined; further rotation of the fibre caused the "blip" to reappear, but in the other liquid*.

TABLE I

Angle of Contact of Wool Fibres at an Oil–Water Interface

Fibre	Contact angle (°)				Mean contact angle (°)
Crossbred slubbing	137	135	140	142	140
	135	145	140	147	
Tippy-dyeing loose wool (dyed with 1% Coomassie Blue RLS)					
(*a*) Dyed areas	90	90	90	90	90
	90	90	90	90	
(*b*) Undyed areas	117	130	125	120	122
	128	120	125	120	
Tippy-dyeing loose wool (dyed with 1% Procion Brilliant Red 5BS)					
(*a*) Dyed areas	100	102	90	95	95
	95	90	95	95	
(*b*) Undyed areas	130	130	125	130	130
	135	130	130	130	

The crossbred slubbing and the undyed areas of the tippy-dyeing loose wool both had high contact angles, indicating that the surfaces were strongly hydrophobic. On the other hand, the dyed areas of the tippy loose wool made a contact angle of approx. 90°, i.e. the fibre was on the point of becoming hydrophilic. The small difference in the contact angles of the undyed areas of wool dyed with Coomassie Blue RL and of wool dyed with Procion Brilliant Red 5B probably results from differences in the dyeing conditions.

At this stage, to provide controls, contact-angle measurements were made on viscose rayon, Terylene, and polypropylene fibre. The results are given in Table II.

TABLE II

Contact Angles of Fibres other than Wool at an Oil–Water Interface

Fibre	Mean fibre diameter (μ)	Treatment	Mean contact angle (°)
Viscose rayon	48·2	—	5
Terylene	20·8	—	150
Terylene	20·8	2% NaOH, 0·2% Lissolamine A50% 30 min at 100°C	90
Polypropylene	27·2	—	175

These results show very clearly the extremely hydrophobic character of polypropylene fibre, which is even greater than that of Terylene fibre. This, in turn, is more hydrophobic than wool fibre, as shown by Table I. It will also be noticed that the undamaged portions of the wool fibre surface are approaching Terylene in hydrophobic properties. On the other hand, the almost completely hydrophilic character of viscose rayon is shown by its very low contact angle. Treating Terylene fibre at the boil with caustic soda makes its surface much less hydrophobic, and the angle of contact is identical with that of the dyed portions of tippy-dyeing loose wool given in Table I. This effect results from the generation of hydroxyl groups on the fibre surface.

Effect of Chlorination and Caustic Potash–Butanol

The effect on wool of chlorination and of treatment with caustic potash–butanol, which are known to have profound effects on dyeing behaviour, was next investigated. Samples of the solvent-extracted crossbred slubbing were treated as follows.

* It should be noted that there are two angles of contact, the values depending on whether the liquid is tending to advance over a dry surface (advancing contact angle) or to recede from a previously wetted one (receding contact angle). In these experiments, the angle was always measured with the fibres receding from the oil and advancing into the water. This gave a clearer and better defined angle than when the fibres were moved in the opposite direction.

(*a*) *Chlorination*—Wool was treated for 15 min at 15°C at a 25:1 liquor ratio in a solution containing 3 ml/l. hydrochloric acid (32°Tw) and 6 ml/l. sodium hypochlorite solution (15% available chlorine). The material was removed, squeezed, and given an anti-chlor for 10 min at 40°C in a 0·3% solution of sodium sulphite, after which it was rinsed and dried.

(*b*) *Treatment with KOH–butanol*—A solution was prepared by heating caustic potash (12 g) in butanol (100 ml) on a water bath. White spirit (100 ml) was then added to give a 6% solution of caustic potash in butanol–white spirit. Extracted crossbred slubbing (8 g) was immersed in this solution (60 ml) at 15°C for 0·5, 1, 2, 4 and 8 min. The material was removed from the solution, squeezed by hand, and immediately plunged into a 5% solution (by volume) of sulphuric acid (168°Tw) (60 ml) and allowed to stand for 5 min in the cold, rinsed in distilled water until neutral, and dried.

Contact angles were then determined, the results being detailed in Table III.

The effects of these treatments on the fibre surface are immediately obvious, chlorination converting a hydrophobic fibre into one which is strongly hydrophilic. Treatment with KOH–butanol–white spirit is clearly progressive. Treatment for 0·5 or 1 min yields a fibre which is neither hydrophobic nor hydrophilic; treatment for 2 min yields a slightly hydrophilic fibre, and further treatment results in a strongly hydrophilic fibre surface similar to that obtained on chlorination.

TABLE III

Contact Angles of Treated Wool Fibres at an Oil–Water Interface

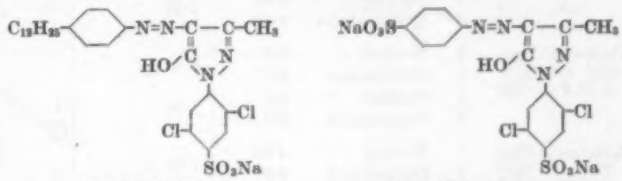
Sample	Mean contact angle (20 measurements) (°)
Untreated slubbing	135
Chlorinated slubbing	28
KOH–butanol–white spirit 0·5 min	90
,, ,, 1 min	90
,, ,, 2 min	70
,, ,, 4 min	30
,, ,, 8 min	30

The results with KOH–butanol–white spirit are interesting in that there is a gradual loss in hydrophobic character. This suggests a gradual erosion of the surface layers and not removal of a discrete film, as might be expected.

DYEING AND PARTITION EXPERIMENTS

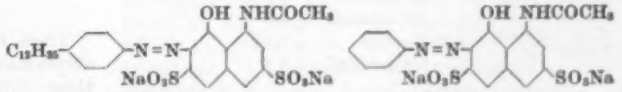
Relation between Constitution of Dye and Tippy-dyeing Behaviour

If differences in the hydrophobic or hydrophilic character of the wool fibre surface are the primary cause of tippy dyeing, this should be clearly shown in the initial dye absorption of dyes of similar constitution but of different hydrophobic character. Therefore, the addition of strongly hydrophobic groups to a dye should result in an increased ability of the dye to diffuse through the hydrophobic surface layers and should thereby reduce differential dyeing behaviour on tippy- or skittery-dyeing wools. To verify this, the initial absorption of two pairs of dyes on wool slubbing and chlorinated wool slubbing was determined, the chlorinated wool being used to represent the tip or damaged portions of the wool. (The results listed in Table III show that acidic chlorination does produce a hydrophilic wool surface.) The dye pairs were—



Carbolan Yellow 3G (C.I. Acid Yellow 72) — Lissamine Fast Yellow 2G (C.I. Acid Yellow 17)

(*a*)



Carbolan Crimson B (C.I. Acid Red 138) — Azo Geranine 2G (C.I. Acid Red 1)

(*b*)

Samples (4 g) of both untreated and chlorinated cross-bred slubbing prepared as described earlier were dyed in the same bath. The material was entered at 40°C into a bath containing, for dyeing with Carbolan dyes, 3% ammonium acetate and 10% Glauber's salt, and, for dyeing with level-dyeing acid dyes, 3% sulphuric acid and 10% Glauber's salt. The temperature was raised to 100°C in 30 min, and the solution boiled for 15 min, by which time equilibrium between fibre and acid had been attained. Dye (1%) was then added and dyeing allowed to take place for either 1 or 2 min. The material was removed, rinsed and dried, and the dye present estimated colorimetrically on a Spekker colorimeter after being Soxhlet-extracted with 25% aqueous pyridine.

A short-time dyeing period was used, because these experiments were designed to find out what happens in the early stages of dyeing. Clearly, the results will not necessarily bear any relation to those obtained under practical dyeing conditions, where, during boiling, migration of dye, particularly of level-dyeing acid dyes, can mask any effects obtained initially. The results of these experiments are detailed in Table IV.

With the yellow dyes, the ratio (dye on chlorinated wool)/(dye on normal wool) is smaller in the initial stages of dyeing for the Carbolan dye than for the analogous level-dyeing acid dye. The presence of the hydrophobic alkyl group in place of a sulphonic group in Carbolan Yellow 3G does, therefore, result in a marked decrease in the difference in the amounts of dye absorbed into normal and into chlorinated wool, presumably because Carbolan Yellow 3G can penetrate more readily through the hydrophobic surface of the unchlorinated fibre. As the Carbolan dyes are of

TABLE IV

Initial Adsorption of Dye on Equal Weights of Untreated and Chlorinated Crossbred Slubbing at 100°C

Dye	Dyeing time (min)	Fibre	Dye on fibre (mg/g wool)	Ratio (Dye on chlorinated wool/Dye on normal wool)
Azo Geranine 2GS (C.I. Acid Red 1)	1	Normal	5·3	1·77
	1	Chlorinated	9·4	
	2	Normal	6·15	1·57
	2	Chlorinated	9·65	
Carbolan Crimson BS (C.I. Acid Red 138)	1	Normal	3·8	1·61
	1	Chlorinated	6·1	
	2	Normal	4·8	1·44
	2	Chlorinated	6·9	
Lissamine Fast Yellow 2GS (C.I. Acid Yellow 17)	1	Normal	4·65	1·98
	1	Chlorinated	9·2	
	2	Normal	6·25	1·80
	2	Chlorinated	11·3	
Carbolan Yellow 3GS (C.I. Acid Yellow 72)	1	Normal	3·6	1·05
	1	Chlorinated	3·8	
	2	Normal	4·15	1·17
	2	Chlorinated	4·8	

higher molecular weight than the level-dyeing acid dyes, it is to be expected that their rates of diffusion through wool will be lower; the importance of these experiments lies only in the ratio, or relative effect.

Of the red dyes, Carbolan Crimson B is not much more uniformly absorbed than Azo Geranine 2G. The only explanation offered is that Carbolan Crimson B is not, in fact, as hydrophobic as would be expected from consideration of its chemical structure. An empirical measure of the hydrophobic character of a dye can be obtained by measuring at room temperature its partition between water and a water-immiscible organic solvent. Butyl alcohol was used for a series of partition tests, as less polar organic solvents gave too low a solubility of dye in the solvent to allow easy measurement. The results obtained are given in Table V.

TABLE V

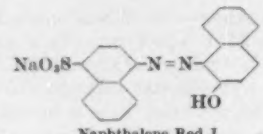
Partition of Dye between Butyl Alcohol and Water

Dye	Partition Ratio (Butyl Alcohol:Water)*
Lissamine Fast Yellow 2G	0·06
Carbolan Yellow 3G	55·0
Azo Geranine 2G	0·02
Carbolan Crimson B	0·68

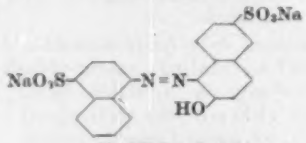
* Based on amount of wool (liquor ratio 25:1), using 3% sulphuric acid with level-dyeing acid dyes and 3% ammonium acetate with Carbolan dyes.

It is clear that Carbolan Crimson B is not as hydrophobic as Carbolan Yellow 3G, although it is a little more hydrophobic than Lissamine Fast Yellow 2G and Azo Geranine 2G. If the results in Table IV and Table V are taken together, they provide good confirmation of the importance of the hydrophobic nature of the dye in determining tippy-dyeing behaviour.

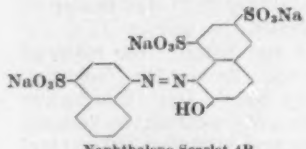
Further examples are provided by the dyes—



Naphthalene Red J
(C.I. Acid Red 88)



Naphthalene Red EA
(C.I. Acid Red 13)



Naphthalene Scarlet 4R
(C.I. Acid Red 18)

These dyes are identical except in the degree of sulphonation. It is well known that the ability of these dyes to give level dyeings on tippy-dyeing wools or on unevenly chlorinated wools decreases as the degree of sulphonation increases. Partition of these dyes between butyl alcohol and water has been determined; the results are given in Table VI. The last column in Table VI is the ratio of the amount of dye absorbed, after 5 min dyeing at 80°C, on chlorinated wool to that on normal wool dyed in the same bath. These results were obtained by Townend and Simpson [22].

TABLE VI

Dye	Partition Ratio (Butyl Alcohol:Water)*	Ratio (Dye on chlorinated wool/dye on normal wool) at 80°C
4·7% Naphthalene Red JS	132	2·6
5·0% Naphthalene Red EAS	2·2	16·6
7·5% Naphthalene Scarlet 4RS	0·058	24·4

* Based on amount of wool using liquor ratio 25:1 and 3% sulphuric acid.

There is excellent correlation between the hydrophobicity of the three dyes (as measured by the reciprocal of the butyl alcohol:water partition ratio) and their ability to dye uniformly on normal and chlorinated wool.

The level-dyeing acid dyes have been classified [23, 24] according to their ability to cover unlevel-dyeing wools. The partition between butyl alcohol and water of level-dyeing acid dyes has been determined; the results are summarised in Fig. 2. As expected, from the previous discussion, there is good correlation between ability to cover uneven-dyeing wool and the hydrophobic properties as determined by partition.

Previous workers [23, 24] have drawn attention to the relation between degree of sulphonation and ability to cover uneven-dyeing wools. The reason

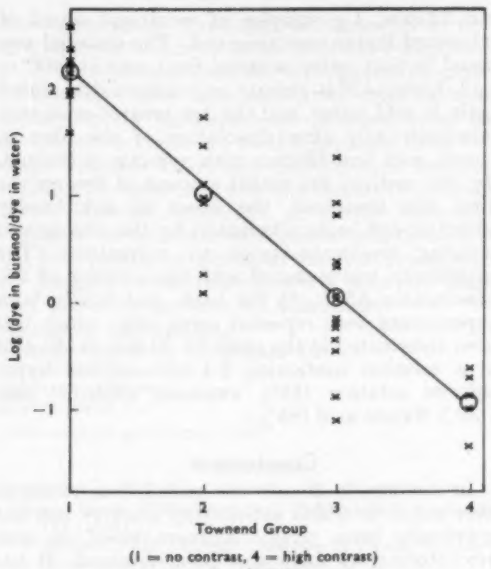


FIG. 2

for this is now clear. The molecular weights of level-dyeing acid dyes lie between fairly narrow limits. If the dye molecule is considered without the sulpho groups, little error will be introduced if it is assumed that the interfacial energy between the unsulphonated level-dyeing acid dye structure and the water will be approximately equal for all dyes. Therefore, the hydrophobic properties of the dye, which determine the ability to cover tippy wool, will depend upon the degree of sulphonation, which is reflected in the partition ratio between butyl alcohol and water.

If milling acid dyes are considered, no simple relation is observed between ability to cover uneven-dyeing wools and degree of sulphonation. With milling acid dyes, which vary widely in molecular weight, the dyeing properties on uneven-dyeing wools will depend on two factors— degree of sulphonation and molecular weight. Clearly, an increase in degree of sulphonation will increase the hydrophilic properties of the dye and thereby produce more uneven dyeing. Increase in molecular weight will have two effects. An increase resulting from an increase in the hydrophobic portion of the dye molecule will enhance the ability of the dye to cover uneven-dyeing wools. Barritt and Elsworth[24] have stated in this connection that Carbolan dyes give less contrast on chlorinated and unchlorinated wools than other milling acid dyes. An increase in molecular weight will also lead to increased steric hindrance to diffusion and will thereby tend to give more unlevel dyeing; this effect will not be large until the molecular size approaches a critical value.

Relation between Dyeing Conditions and Tippy-dyeing Behaviour

1:1 Metal-complex dyes, e.g. Ultralans, require large quantities of sulphuric acid to prevent their giving very tippy dyeings. No satisfactory explanation of this behaviour has been given. Measurements were therefore made of the partition of Ultralan Pink BN (C.I. Acid Red 186) between butyl alcohol and water, and an attempt was made to compare these results with an assessment of tippy-dyeing behaviour. The results are shown in Table VII.

TABLE VII

Partition and Dyeing Behaviour of Ultralan Pink BNS

Sulphuric acid concentration (%)	Partition Ratio (Butyl alcohol: Water)*	Tippy-dyeing behaviour (1 = Poor 5 = Excellent)
0	0·45	—
1	1·71	1
3	4·32	2
5	6·00	3
8	7·47	5
12	8·11	5

* Water phase calculated on liquor ratio = 25:1; [dye] = 1·0%.

Table VII clearly shows that an increase in the concentration of sulphuric acid in the dyebath increases the hydrophobic behaviour of the dye, which will allow more ready diffusion through the epicuticle. It is therefore not surprising that good correlation is again found between covering of tippy-dyeing wool and partition between butyl alcohol and water.

Mode of Action of Auxiliary Products

Certain auxiliary products can reduce the tippy dyeing of wool. It was thought that, in all cases, these products form a complex with the dye and thereby increase its hydrophobic properties. The results of a series of partition experiments are given in Table VIII.

TABLE VIII

Partition of Ultralan Pink BNS (1%) in presence of Lissolamine A50%* (2%)

Sulphuric acid concentration (%)	Partition Ratio (Butyl alcohol: water)†
0	4·6
1	5·7
3	8·4
5	9·2
8	8·0
12	7·8

* Lissolamine A— cetyltrimethyl ammonium bromide.
† Liquor ratio 25:1; all amounts calculated as if fibre were present.

When the results in Table VII are compared with those in Table VIII, the increase in hydrophobic character of the dye–agent complex is clearly seen. If, with Ultralan Pink BN, as the results from Table VII suggest, a partition ratio of 7·5 (using 8% of sulphuric acid) results in good coverage of tippy-dyeing wool, the same effect should be obtained with 2% of Lissolamine A50% and only 3% of sulphuric acid. Dyeing tests have confirmed this. It is not suggested that dyeing Ultralan dyes from a dyebath containing 3% of sulphuric acid and 2% of Lissolamine A50% is a practical dyeing process, as there are defects in the method, resulting mainly from precipitation of the dye–agent complex. The observations of Rattee[25]

TABLE IV

Initial Adsorption of Dye on Equal Weights of Untreated and Chlorinated Crossbred Slubbing at 100°C

Dye	Dyeing time (min)	Fibre	Dye on fibre (mg/g wool)	Ratio (Dye on chlorinated wool/Dye on normal wool)
Azo Geranine 2GS (C.I. Acid Red 1)	1	Normal	5·3	1·77
	1	Chlorinated	9·4	
	2	Normal	6·15	1·57
	2	Chlorinated	9·65	
Carbolan Crimson B8 (C.I. Acid Red 138)	1	Normal	3·8	1·61
	1	Chlorinated	6·1	
	2	Normal	4·8	1·44
	2	Chlorinated	6·9	
Lissamine Fast Yellow 2GS (C.I. Acid Yellow 17)	1	Normal	4·65	1·98
	1	Chlorinated	9·2	
	2	Normal	6·25	1·80
	2	Chlorinated	11·3	
Carbolan Yellow 3GS (C.I. Acid Yellow 72)	1	Normal	3·6	1·05
	1	Chlorinated	3·8	
	2	Normal	4·15	1·17
	2	Chlorinated	4·8	

higher molecular weight than the level-dyeing acid dyes, it is to be expected that their rates of diffusion through wool will be lower; the importance of these experiments lies only in the ratio, or relative effect.

Of the red dyes, Carbolan Crimson B is not much more uniformly absorbed than Azo Geranine 2G. The only explanation offered is that Carbolan Crimson B is not, in fact, as hydrophobic as would be expected from consideration of its chemical structure. An empirical measure of the hydrophobic character of a dye can be obtained by measuring at room temperature its partition between water and a water-immiscible organic solvent. Butyl alcohol was used for a series of partition tests, as less polar organic solvents gave too low a solubility of dye in the solvent to allow easy measurement. The results obtained are given in Table V.

TABLE V

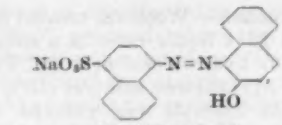
Partition of Dye between Butyl Alcohol and Water

Dye	Partition Ratio (Butyl Alcohol:Water)*
Lissamine Fast Yellow 2G	0·06
Carbolan Yellow 3G	55·0
Azo Geranine 2G	0·02
Carbolan Crimson B	0·68

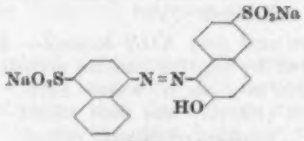
* Based on amount of wool (liquor ratio 25 : 1), using 3% sulphuric acid with level-dyeing acid dyes and 3% ammonium acetate with Carbolan dyes.

It is clear that Carbolan Crimson B is not as hydrophobic as Carbolan Yellow 3G, although it is a little more hydrophobic than Lissamine Fast Yellow 2G and Azo Geranine 2G. If the results in Table IV and Table V are taken together, they provide good confirmation of the importance of the hydrophobic nature of the dye in determining tippy-dyeing behaviour.

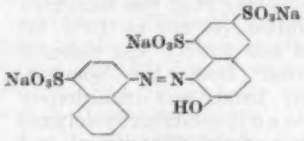
Further examples are provided by the dyes—



Naphthalene Red J
(C.I. Acid Red 88)



Naphthalene Red EA
(C.I. Acid Red 13)



Naphthalene Scarlet 4R
(C.I. Acid Red 18)

These dyes are identical except in the degree of sulphonation. It is well known that the ability of these dyes to give level dyeings on tippy-dyeing wools or on unevenly chlorinated wools decreases as the degree of sulphonation increases. Partition of these dyes between butyl alcohol and water has been determined; the results are given in Table VI. The last column in Table VI is the ratio of the amount of dye absorbed, after 5 min dyeing at 80°C, on chlorinated wool to that on normal wool dyed in the same bath. These results were obtained by Townend and Simpson [22].

TABLE VI

Dye	Partition Ratio (Butyl Alcohol:Water)*	Ratio (Dye on chlorinated wool/dye on normal wool) at 80°C
4·7% Naphthalene Red JS	132	2·6
5·0% Naphthalene Red EAS	2·2	16·6
7·5% Naphthalene Scarlet 4RS	0·058	24·4

* Based on amount of wool using liquor ratio 25 : 1 and 3% sulphuric acid.

There is excellent correlation between the hydrophobicity of the three dyes (as measured by the reciprocal of the butyl alcohol:water partition ratio) and their ability to dye uniformly on normal and chlorinated wool.

The level-dyeing acid dyes have been classified [23, 24] according to their ability to cover unlevel-dyeing wools. The partition between butyl alcohol and water of level-dyeing acid dyes has been determined; the results are summarised in Fig. 2. As expected, from the previous discussion, there is good correlation between ability to cover uneven-dyeing wool and the hydrophobic properties as determined by partition.

Previous workers [23, 24] have drawn attention to the relation between degree of sulphonation and ability to cover uneven-dyeing wools. The reason

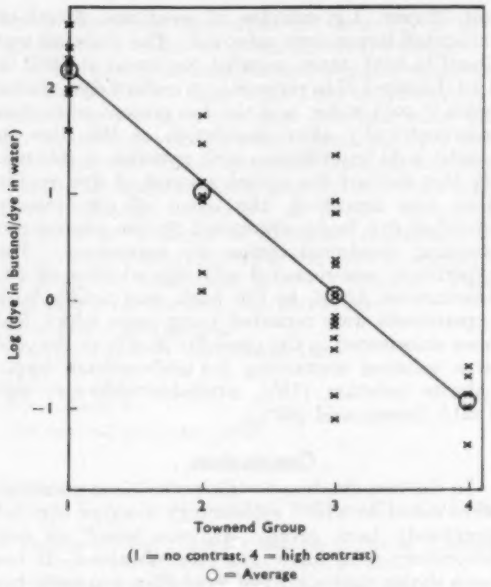


FIG. 2

for this is now clear. The molecular weights of level-dyeing acid dyes lie between fairly narrow limits. If the dye molecule is considered without the sulpho groups, little error will be introduced if it is assumed that the interfacial energy between the unsulphonated level-dyeing acid dye structure and the water will be approximately equal for all dyes. Therefore, the hydrophobic properties of the dye, which determine the ability to cover tippy wool, will depend upon the degree of sulphonation, which is reflected in the partition ratio between butyl alcohol and water.

If milling acid dyes are considered, no simple relation is observed between ability to cover uneven-dyeing wools and degree of sulphonation. With milling acid dyes, which vary widely in molecular weight, the dyeing properties on uneven-dyeing wools will depend on two factors— degree of sulphonation and molecular weight. Clearly, an increase in degree of sulphonation will increase the hydrophilic properties of the dye and thereby produce more uneven dyeing. Increase in molecular weight will have two effects. An increase resulting from an increase in the hydrophobic portion of the dye molecule will enhance the ability of the dye to cover uneven-dyeing wools. Barritt and Elsworth[24] have stated in this connection that Carbolan dyes give less contrast on chlorinated and unchlorinated wools than other milling acid dyes. An increase in molecular weight will also lead to increased steric hindrance to diffusion and will thereby tend to give more unlevel dyeing; this effect will not be large until the molecular size approaches a critical value.

Relation between Dyeing Conditions and Tippy-dyeing Behaviour

1:1 Metal-complex dyes, e.g. Ultralans, require large quantities of sulphuric acid to prevent their giving very tippy dyeings. No satisfactory explanation of this behaviour has been given. Measurements were therefore made of the partition of Ultralan Pink BN (C.I. Acid Red 186) between butyl alcohol and water, and an attempt was made to compare these results with an assessment of tippy-dyeing behaviour. The results are shown in Table VII.

TABLE VII

Partition and Dyeing Behaviour of Ultralan Pink BNS

Sulphuric acid concentration (%)	Partition Ratio (Butyl alcohol: Water)*	Tippy-dyeing behaviour (1 = Poor 5 = Excellent)
0	0·45	—
1	1·71	1
3	4·32	2
5	6·00	3
8	7·47	5
12	8·11	5

* Water phase calculated on liquor ratio = 25:1; [dye] = 1·0%.

Table VII clearly shows that an increase in the concentration of sulphuric acid in the dyebath increases the hydrophobic behaviour of the dye, which will allow more ready diffusion through the epicuticle. It is therefore not surprising that good correlation is again found between covering of tippy-dyeing wool and partition between butyl alcohol and water.

Mode of Action of Auxiliary Products

Certain auxiliary products can reduce the tippy dyeing of wool. It was thought that, in all cases, these products form a complex with the dye and thereby increase its hydrophobic properties. The results of a series of partition experiments are given in Table VIII.

TABLE VIII

Partition of Ultralan Pink BNS (1%) in presence of Lissolamine A50%* (2%)

Sulphuric acid concentration (%)	Partition Ratio (Butyl alcohol: water)†
0	4·6
1	5·7
3	8·4
5	9·2
8	8·0
12	7·8

* Lissolamine A— cetyltrimethyl ammonium bromide.
† Liquor ratio 25:1; all amounts calculated as if fibre were present.

When the results in Table VII are compared with those in Table VIII, the increase in hydrophobic character of the dye–agent complex is clearly seen. If, with Ultralan Pink BN, as the results from Table VII suggest, a partition ratio of 7·5 (using 8% of sulphuric acid) results in good coverage of tippy-dyeing wool, the same effect should be obtained with 2% of Lissolamine A50% and only 3% of sulphuric acid. Dyeing tests have confirmed this. It is not suggested that dyeing Ultralan dyes from a dyebath containing 3% of sulphuric acid and 2% of Lissolamine A50% is a practical dyeing process, as there are defects in the method, resulting mainly from precipitation of the dye–agent complex. The observations of Rattee[25]

on dyeing with 1:1 metal-complex dyes in presence of small amounts of sulphuric acid and Lissapol N can probably also be explained in terms of the formation of hydrophobic dye–Lissapol N complexes.

Explanation of Rate of Dyeing Phenomenon

It has recently been reported[26,27] that Cibacron Scarlet 2G (C.I. Reactive Red 9) dyes wool more rapidly in the presence of cationic agents. This observation has not been adequately explained and at first sight seems very surprising, as it might be expected that the complex formed, being of higher molecular weight, would dye more slowly. However, the ideas developed in this paper indicate that a highly sulphonated dye, such as Cibacron Scarlet 2G, cannot readily diffuse across the hydrophobic epicuticle of wool, whereas the more hydrophobic dye–cationic agent complex can. In effect, therefore, there is a larger effective surface available for the dye–agent complex to diffuse across, and therefore the increase in rate of dyeing is understandable. With chlorinated wool, where some of the epicuticle has been removed, any increase in rate of dyeing caused by the addition of a cationic agent would be expected to be less marked. The experimental results in Fig. 3 obtained with Procion Scarlet H3GS (C.I. Reactive Red 9) confirm this observation and also indicate, as predicted, that the effect is less pronounced on chlorinated wool. The experimental technique was as follows.

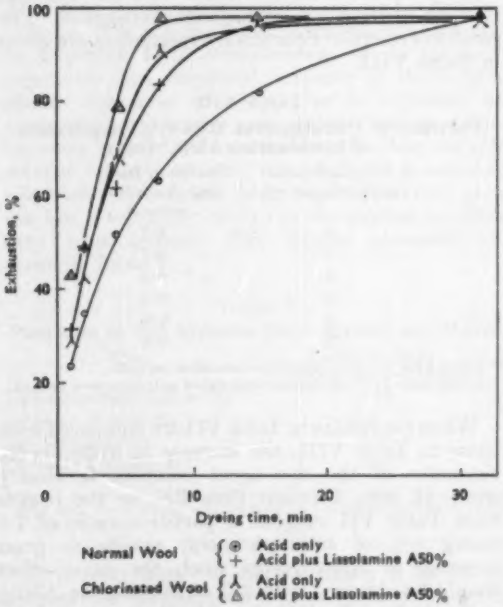


FIG. 3—Effect of Lissolamine A50% on the rate of dyeing of Procion Scarlet H3GS at 100°C

A dyebath was prepared at 100°C containing 1% Procion Scarlet H3GS and 5% acetic acid (30%), at a 50:1 liquor ratio. Wetted-out wool was introduced and, after periods of 1, 2, 4, 8, 16 and 32 min, 1-g samples of wool and 50 ml of exhausted liquor were removed. The material was rinsed in cold water, scoured for 1 min at 50°C in 3 g/l. Lissapol N to remove any unfixed dye, rinsed again in cold water, and the dye present estimated colorimetrically after dissolution of the fibre in caustic soda and dilution with pyridine or ethanol. By this method the actual amount of dye on the fibre was measured, the effect of any loosely adsorbed dye being eliminated by the rinsing and scouring treatment prior to extraction. The experiment was repeated with the addition of 2% Lissolamine A50% to the bath, and finally both experiments were repeated using serge which had been chlorinated in the piece for 30 min in the cold in a solution containing 3·4 ml/l. sodium hypochlorite solution (15% available chlorine) and 1 ml/l. formic acid (85%).

Conclusions

In the introduction to this paper, four questions were asked to which satisfactory answers had not previously been given. Answers based on new laboratory work have now been obtained. It has been shown that a normal wool fibre generally has a hydrophobic surface, except where the fibre is damaged, when the surface becomes more hydrophilic. During the initial period of dyeing, before subsequent levelling of dye can reduce the effect, the absorption of dye by the wool fibre can be very greatly influenced by the hydrophobic nature of the fibre surface. Initial absorption also depends upon the properties of the dye. If the dye is very hydrophilic, differences in the hydrophobic properties of the fibre surface are reflected in differences in dye absorption. For this reason hydrophilic dyes are tippy dyeing. If the dye is more hydrophobic, it can diffuse more readily through the hydrophobic surface layers of the fibre, where these are present, and there will, as a result, be less contrast between the hydrophilic and hydrophobic portions of the wool surface. For this reason hydrophobic dyes are less tippy dyeing.

The influence of chemical constitution and dyeing techniques on tippy dyeing has been studied, and the results obtained provide further confirmation of the explanation outlined above. In addition, the results also permit a more satisfactory explanation of the effect of degree of sulphonation of the dye and the mode of action of cationic auxiliary products. In both these cases, the importance of the hydrophobic nature of the wool fibre surface and the balance of the hydrophobic–hydrophilic properties of the dye or dye–cationic agent complex is shown. Although attention has been concentrated on level-dyeing acid dyes, the same concepts have been shown to apply to 1:1 premetallised, reactive, and milling acid dyes. No work has been done on 1:2 premetallised dyes but, in a general way, it can be seen that their dyeing behaviour will fall into the same general pattern and, in fact, it can now be seen more clearly why, in spite of the large molecular size of these dyes, they give good coverage of tippy-dyeing wool. Schetty[29] has emphasised the necessity of reducing

the hydrophilic character of these dyes to a minimum in order to avoid skittery dyeings.

In the work described, the hydrophobic properties of the dye have been estimated empirically by measuring the partition ratio between water and an organic solvent. It would, perhaps, have been more desirable if this empirical estimate had been replaced by measurements of a thermodynamically more definable property. A purely theoretical study of the influence of chemical constitution on the hydrophobic properties of the dyes, as suggested by Meggy[28], would also prove of interest. These are, however, matters for the future.

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(*MS. received 5th October 1960*)

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Discussion

MIDLANDS SECTION

Mr. S. M. JAECKEL: Why does Ultralan Pink BNS in presence of sulphuric acid give an increasing partition ratio between butanol and water as the acid concentration increases, whereas when Lissolamine A is present as well the partition ratio appears to reach a maximum with 5% sulphuric acid? Secondly, where dyes become less skittery dyeing as they become more hydrophobic, does the addition of wetting agent tend to make the system more hydrophilic?

Mr. LEMIN: There appears to be no simple explanation for the partition maximum in the presence of Lissolamine A. As far as the second question is concerned, auxiliary products or wetting agents affect the hydrophobic properties of dyes only when they form complexes. A wetting agent which does not complex with the dye will not affect the hydrophobic properties of the dye and hence its dyeing properties, although the aqueous phase of the system might be rendered more hydrophilic.

Mr. J. WALTON: Is there any relation between the hydrophobic or hydrophilic properties of the wool fibre surface and the ortho- or para-cortical structure of the fibre?

Mr. HADFIELD: Not so far as we are aware.

Mr. WALTON: Is it implied in the paper that level-dyeing acid dyes cover tippy wool less well than acid-milling dyes?

Mr. HADFIELD: The results presented refer to the initial stages of the dyeing process only. In these early stages of dyeing the more hydrophobic acid-milling dyes were adsorbed more uniformly than the hydrophilic level-dyeing acid dyes. However, during a normal dyeing process, where, in boiling, level-dyeing acid dyes migrate well and milling dyes very little, the initial uneven adsorption of level-dyeing acid dyes would be overcome, but little change would be expected with milling dyes. Thus, under normal dyeing conditions a final level dyeing would be expected with most level-dyeing acid dyes, whereas many milling dyes would still show tippiness.

Dr. W. L. LEAD: Why was butanol chosen as the solvent in the partition experiments? Would the results of the present work have any relevance for solvent dyeing? Secondly, does caustic soda have a specific action on the wool surface resulting in an altered surface nature?

Mr. HADFIELD: Butanol was chosen in preference to other solvents in the partition experiments simply because it gave a very convenient spread of results, and not for any connection with solvent dyeing. However, the work helped to throw light on the subject of solvent dyeing. The function of butanol or other agents in solvent dyeing was suggested as being that of a "carrier" for the dye across the epicuticle. The effect of solvents on hydrophilic dyes was very small and they had no effect on fibres free from epicuticle (i.e. regenerated proteins or chlorinated wool).

In reply to the second question, caustic soda acts on the fibre partly by epicuticle breakdown, but no precise information is available on the exact mechanism.

Mr. D. B. WILLIAMSON: If the hydrophilic properties of a dye could be correlated with molecular weight, could dye solubility be correlated with the problem being investigated?

Mr. LEMIN: There is a very rough correlation between solubility of dye and the initial covering of tippy wool, but exceptions can be found. These exceptions are mainly aggregated dyes where the solubility can be considered as being midway between true solubility and ultrafine dispersion. Disperse dyes and almost insoluble chrome dyes (Solochrome Red DS or Solochrome Brown MGS) give excellent covering of tippy wool.

Mr. J. C. HAWLEY: Lissolamine A has an offensive smell; have anionic agents been investigated as blocking auxiliaries?

Mr. HADFIELD: We agree that Lissolamine A has an unpleasant smell; however, in the experimental work we were concerned only with an academic approach to the problem. The effect of anionic agents in emphasising the tippy-dyeing characteristics of wool can be understood clearly as a result of this work. They are generally high-molecular-weight compounds of low sulphonation, and in an acid dyebath are adsorbed on to the hydrophobic portions of the fibre. Thus, the more accessible hydrophilic parts of the fibre only are originally available for dye adsorption.

Mr. A. SHENTON: What is the effect of shrink-resist processes on the wool fibre surface? I understand that the Wolsey–Stevenson process acts differently from other methods in not removing the outer surface layers.

Mr. LEMIN: Most shrink-resist processes modify the epicuticle of wool and hence allow easier penetration of all dyes into the fibre. With processes which give rise to weaker equilibrium dyeings, this is an effect on the acid- and base-binding capacity of the fibre and not an effect on initial rate of dyeing, for even with this type of fibre it has been found that the initial strike of dye is higher than on untreated wool, which indicates some modification of the fibre surface.

COMMUNICATION

Investigations into the Continuous Dyeing of Wool

B. MILLIGAN

A method of dyeing wool at room temperature has been studied, and adapted to the continuous dyeing of slubbing. Formic acid is used as the dye solvent. The method causes negligible fibre damage, and dye penetration and fastness of the dyeings are satisfactory. However, most dyes give skittery dyeings under the conditions used, and satisfactory fixation is obtained only in pale to medium shades. Methods of recovery of the dye solvent are described, and the cost of dyeing is estimated.

Introduction

In a recent letter, Harrap [1] briefly described a process for dyeing wool at room temperature within a few minutes, using anhydrous formic acid as the dye solvent. The method is due to Weller [2], who used formic acid in concentrations of up to 85%*. Other patents were subsequently granted to Martin [3], who used 10–50% formic acid as the dye solvent for both printing and dyeing.

Harrap [1] believes that the effectiveness of formic acid is due to several factors. The rapid wetting and high degree of swelling of wool fibres in formic acid are thought to assist rapid diffusion of dye into the fibres, while the physical properties of formic acid are such that the electrostatic forces of attraction between the basic groups of wool and any sulphonic acid groups in the dye molecules are at a maximum. Contrary to his earlier supposition, Harrap [4] has shown by molecular-weight determinations, using the light-scattering technique, that some acid dyes are more highly aggregated in formic acid than in water. However, the high degree of swelling of the fibres exerts a compensating effect which apparently is sufficient to allow rapid diffusion of the dye aggregates into the fibres.

The rapidity of the process suggested that it might find application in the continuous dyeing of wool, but it was first necessary to ensure that the dyeings produced were of satisfactory fastness, and also that no serious damage to the wool occurred during treatment with formic acid, or during its subsequent removal. Moreover, the cost of this new dyeing process in comparison with that of existing dyeing methods required consideration.

* Formic acid concentrations are expressed on a weight/weight basis throughout.

Fastness and Damage Tests

Table I shows the fastness ratings of dyeings (1%) on Merino 64s slubbing when tested by S.D.C. Fastness to Washing Test No. 2, and by the light-fastness test described in B.S. 1006 : 1955.

The dyeings were prepared as follows—

FORMIC ACID PROCESS

The slubbing was immersed in a solution of dye in 90% formic acid (liquor to wool ratio 15:1) for 2 min at room temperature, and then rinsed in running water for 1 h. The concentration of the dye solution was adjusted so that the depth of shade obtained was approximately 1%; the dyebath was only partly exhausted in every case. Prolonging the immersion time beyond 2 min had no significant effect on the degree of exhaustion.

CONVENTIONAL METHODS

(*a*) Lissamine and Neolan dyes were applied from a dyebath containing 1% dye, 4% sulphuric acid and 20% Glauber's salt at a liquor ratio of 50:1*. The wool was introduced at 40°C, the temperature raised to the boiling point over ½ h, and boiling continued for 1 h. The samples were removed and rinsed in running water for 1 h.

(*b*) Coomassie dyes were applied similarly, 4% acetic acid (30%) being used instead of sulphuric acid.

(*c*) Cibalan and Cibalan Brilliant dyes were applied from a dyebath containing 4% ammonium acetate. The wool was introduced at room temperature, and the temperature raised to the boiling point over ½ h, and kept there for a further hour. The samples were rinsed for 1 h.

* Percentages of dyes and additives are based on weight of wool.

TABLE I
Comparative Fastness Tests

Dye	*Colour Index* No.	Dyeing process*	S.D.C. No. 2 Wash test			Light fastness
			Effect on dyeing	Staining of wool	Staining of cotton	
Lissamine Fast Yellow 2G	Acid Yellow 17	C	2–3	5	5	6
		F	3	5	5	6
Lissamine Fast Red BG	Acid Red 37 / Acid Red 1	C	2–3	5	4–5	5
		F	4	5	4–5	5
Lissamine Fast Red 4G	Acid Red 30	C	2–3	5	5	5
		F	3–4	5	5	5
Coomassie Yellow R	Acid Yellow 42	C	5	5	5	4
		F	5	5	5	4
Coomassie Red PG	Acid Red 85	C	4	5	3	4
		F	4	5	3	4
Coomassie Ultra Sky SE	Acid Blue 112	C	4	5	5	4–5
		F	4	5	5	4
Carbolan Yellow R	Acid Yellow 71	C	5	5	5	5–6
		F	5	5	5	5–6
Carbolan Crimson 3B	Acid Red 139	C	5	5	5	4
		F	5	5	5	4
Carbolan Blue B	Acid Blue 138	C	4	5	5	5
		F	4	5	5	5
Neolan Flavine GFE	Acid Yellow 103	C	2–3	5	5	
		F	3	5	5	
Neolan Red REG†	Acid Red 198	C	3–4	5	5	
		F	5	5	5	
Neolan Green BL†	Acid Green 12	C	3	5	4	
		F	3–4	5	5	
Neolan Blue 2G	Acid Blue 158A	C	5	5	5	
		F	4	5	5	
Cibalan Brilliant Blue GL	Acid Blue 183	C	5	3	5	
		F	5	5	5	
Cibalan Brilliant Red BL	Acid Red 252	C	5	5	5	
		F	4–5	4	5	
Cibalan Brilliant Yellow 3GL	Acid Yellow 127	C	5	5	5	
		F	5	5	5	
Cibacron Scarlet 2G	Reactive Red 9	C	4	5	4	
		F	5	5	3	
Cibacron Brilliant Orange G	Reactive Orange 2	C	5	5	5	
		F	5	5	4	
Cibacrolan Blue 8G†	Acid Blue 185	C	4–5	5	5	
		F	4–5	5	5	

* C = dye applied by conventional method (*a*), (*b*), (*c*) or (*d*).
F = dye applied by formic acid process.
† These dyes gave a brighter shade when applied by method F.

(*d*) Cibacron and Cibacrolan dyes were applied from a dyebath containing 1% dye, 0·5% Neovadine AL, 3% ammonium sulphate, 1% Emulsifier OC and 10% Glauber's salt. The wool was introduced at 40°C, the temperature raised to the boiling point over ¾ h, and kept there for 1 h. The samples were rinsed for 1 h.

Most dyes gave dyeings of similar wash-fastness whether applied by the formic acid process or by conventional methods. The superior wash-fastness of the dyeings obtained with Lissamine and Neolan dyes applied by the formic acid process was shown to be due to the presence of residual formic acid. If the samples were first soaked overnight in 0·5-M phosphate buffer (pH 7), the wash-fastness ratings were then the same as those of dyeings produced by the conventional method.

It was surprising that dyeings of Cibacron and Cibalan Brilliant dyes had similar wash-fastness whether obtained by the conventional method or by the formic acid process, and this suggests that chemical combination of these dyes with wool had taken place even at room temperature in formic acid. This was verified by continuous extraction of the dyeings with hot 80% pyridine. Only a small amount of dye was removed, most of it being resistant to extraction. Pyridine extraction of dyeings obtained with Procion Brilliant Orange G (C.I. Reactive Orange 1), Scarlet G (C.I. Reactive Red 8), Blue HB (C.I. Reactive Blue 2), Brilliant Blue H7G (C.I. Reactive Blue 3), and Brilliant Orange HGR (C.I. Reactive Orange 2) applied by the formic acid process also indicated that most of the dye had combined chemically with the wool.

Dyeings of several of the dyes had the same fastness to light and to rubbing, irrespective of the method of application. The light-fastness ratings are shown in Table I. Carbolan Crimson 3B (C.I. Acid Red 139), Carbolan Blue B (C.I. Acid Red 138), Coomassie Red PG (C.I. Acid Red 85), Coomassie Yellow R (C.I. Acid Yellow 42), Lissamine Fast Red 4G (C.I. Acid Red 30), and

Lissamine Fast Yellow 2G (C.I. Acid Yellow 17) were the dyes used in the rubbing-fastness tests.

Although preliminary tests[1] by a manual-strength testing method[5] indicated that damage caused to wool in the formic acid process was less than that caused in conventional dyeing methods, more precise testing by determining the breaking load of individual fibres has shown that there are no significant differences between the two methods. Dry-combed Corriedale 56s slubbing was dyed to a 1·5% depth of shade with Coomassie Red PG by the formic acid process and by the conventional method (*b*) previously described. The samples are designated F and C, respectively. Other samples were treated in absence of dye, the conditions of dyeing being otherwise identical. These samples are designated FO (formic acid, no dye) and CO (conventional, no dye). The values shown in Table II are the means of 20 determinations of breaking loads of fibres conditioned at 20°C and 65% r.h. Treatment of wool fibres with formic acid alone had no significant effect on the breaking load.

TABLE II

Measurements of Fibre Breaking Load

Treatment	Control	C	F	CO	FO
Breaking strain (g) (Standard error = 1·2)	18·2	15·4	15·3	17·6	16·7

Although it was originally believed that all acid wool dyes and metal-complex dyes could be applied from formic acid solution, it has since been found that some 1 : 2 metal-complex dyes do not develop their normal hue unless the dyeings are subsequently boiled in water for about 10–20 min. Chrome dyes may be applied from a formic acid dyebath in the usual way, but require subsequent chroming in an aqueous bath. Omega Chrome Brown PB (C.I. Mordant Brown 76), Omega Chrome Blue Black B (C.I. Mordant Black 3), Diamond Black PV (C.I. Mordant Black 9), and Eriochrome Red B (C.I. Mordant Red 7) were applied from 90% formic acid solution, the dyebaths being only partly exhausted. The dyeings were rinsed for 5 min, and then boiled for ½ h in an aqueous solution of potassium dichromate (0·5%).

Dye Penetration and Fibre Swelling

Microscopic examination of cross-sections of Merino 64s wool fibres dyed by immersion of loose wool in solutions of acid dyes in 90% and 98% formic acid for 2 min showed that dye penetration was complete. Extension of the dyeing time to about 10 min was necessary to ensure complete penetration of Lincoln wool fibres with milling acid dyes from 90% formic acid solution at room temperature, although treatment for 2 min was sufficient if 98% formic acid was used.

If 70% formic acid is used as the dye solvent, Merino 64s fibres are generally ring-dyed, although complete penetration by level-dyeing acid dyes is possible if the dyeing time is extended to 10 min. However, if the temperature is raised to 40°C, complete penetration by all acid dyes occurs within 2 min, the dyeings so produced being comparable with those obtained using 90% formic acid at room temperature.

In order to determine whether the improvement in dye uptake and penetration obtained on increasing the temperature was related to the degree of swelling of the fibres, measurements of the diameter of Corriedale 56s wool fibres were made over a range of temperatures. Fibre diameters were measured by means of a projection microscope giving an overall magnification of 300. The fibres were mounted singly in a frame which allowed the fibre to be rotated, with provision for clamping the fibre at intervals of 90° rotation. This permitted measurement at four points during a single rotation, and enabled a mean diameter to be determined, errors arising from the selection of fibres of elliptical cross-section being minimised. The frame was constructed so that the fibre rested upon a microscope slide and could be covered by a cover slip. The mean diameter of the untreated fibre was determined at a point chosen at random, a drop of formic acid was introduced under the cover slip, and the mean diameter measured again at the same point, thus affording a value for the increase in fibre diameter. The results are given in Table III where each value is the mean of ten determinations, a different fibre being used for each.

TABLE III

Effect of Temperature and Acid Concentration on Fibre Diameter

Temperature (°C)	Increase in fibre diameter (%) at acid concentration (%) of 98	90	70	Standard error
20–21	69·6	53·0	34·6	1·4
25–26	71·2	54·3	34·8	3·4
39–41	70·2	61·8	42·0	1·6

The temperatures of treatment were achieved by introducing the microscope into a heated chamber covered by a thin polythene sheet in which a slit was cut to permit introduction of the acid and manipulation of the microscope.

The results show that the degree of swelling (as a function of increase in fibre diameter) is highest with 98% acid and lowest with 70% acid, but that at the higher concentration temperature has little effect. However, with both 70% and 90% acid, the degree of swelling is increased by raising the temperature from 20–21°C to 39–41°C. Nevertheless, the degree of swelling produced by 70% acid at 39–41°C is still considerably less than that produced by 90% acid at 20–21°C. Thus it appears that the degree of swelling produced by the dye solvent is not the sole factor determining the rate of dye uptake and penetration, since dye solutions in 70% formic acid at 40°C and in 90% acid at room temperature give similar dyeings. It may be that increase in temperature results in some disaggregation of dye aggregates, and in increased diffusion in solution, thus increasing the rate of dye diffusion into the fibres.

Dyeing Fibres other than Wool

Alpaca, cashmere and mohair may also be dyed with acid dyes in 90% formic acid at room temperature, dye penetration being complete within 2 min, except on mohair. Only partial exhaustion of the dye solution was possible.

Manual strength testing [5] indicated that there had been no fibre damage or adverse effect on the handle. Acrilan, Fibrolane and Merinova can also be dyed satisfactorily, but silk, Vicara and Ardil, although possessing affinity for acid dyes in formic acid solution, suffer considerable loss in strength.

Continuous Dyeing Methods

Because of the relatively high degree of swelling of wool by formic acid, a liquor ratio of at least 15:1 is necessary to ensure complete immersion of the wool during dyeing. The relatively high cost of formic acid and the need to use long liquor ratios thus preclude the use of a batch process. Since the dye liquor is only partially exhausted, the bath could only be used for dyeing a second batch to the same colour and such a procedure would be unsatisfactory. In addition, the time of dyeing is so short that the delay in loading large batches into the bath would almost certainly result in unlevel dyeing.

Since recovery of the formic acid is essential for economical operation of the process, a dyeing method in which the liquor ratio is as low as possible is preferable. A pad-dyeing method was therefore employed, and a small hand-operated padding machine was constructed for preliminary work. Subsequently, another padding machine was used in conjunction with a recovery unit for removing the formic acid from dyed slubbing by countercurrent washing with water.

APPARATUS

Hand-operated Padding Machine

This machine was of the type shown in Fig. 1, and was designed primarily for the continuous padding of slubbing, although it could also be used for pad-dyeing narrow strips of fabric. The squeeze rollers (B, C) were kept under tension by means of two heavy coil springs. A third roller (A) was submerged in the dyebath to keep the slubbing in the dye liquor. Roller C (1-in. diam. × 2½ in.) was constructed of rubber, and rollers A and B of polythene (1½-in. diam. × 2½ in.). Rubber, polythene, stainless steel and brass were all satisfactorily resistant to corrosion by formic acid if the equipment was washed well with water after use. Roller C was driven by means of a crank handle, speeds of throughput of up to 10 yd/min being comfortably maintained.

The distance between the nip of the squeeze rollers and the surface of the dye liquor was found to be important. Continual breakage of the slubbing took place if this distance appreciably exceeded the mean fibre length.

Machine-driven Padding Machine— Washing Train

A diagram of this machine is shown in Fig. 1. The padding unit was essentially the same as that of the hand-operated machine, except that both squeeze rollers (B, C) were of Neoprene rubber (1½-in. diam. × 2½ in.). The washing train consisted of six small wash-bowls (1–6), and a long wash-bowl (7), each fitted with a pair of brass squeeze rollers (1-in. diam. × 2½ in.) and either a roller or frame to keep the slubbing immersed during its passage through the bowl. Each wash-bowl was joined to the next by a weir, so that water passing into bowl 7 eventually flowed out from bowl 1 into a receiver, thus effecting a countercurrent washing of the slubbing. The padded slubbing was held by "skying" before washing so that maximum dye fixation could take place. Roller C and the corresponding seven rollers in the countercurrent washing unit were rotated at the same speed by means of an electric motor, driving the slubbing at the rate of 5 yd/min.

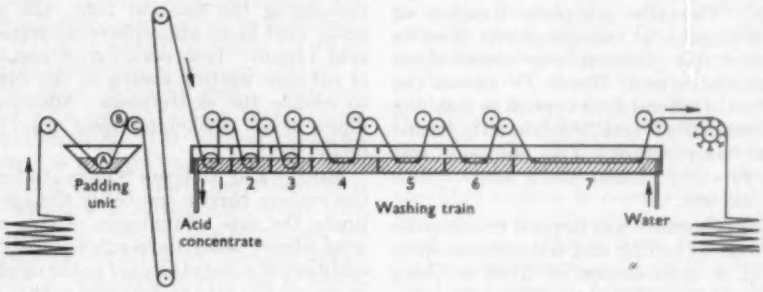

FIG. 1

MATERIAL

The slubbing used had been French-combed; an air-dried sample contained 1·0–1·5% grease (determined by Soxhlet extraction with methylene dichloride).

Fabric A: Plain weave, 31 ends/in., 30 picks/in., weight 7·1 oz/yd (56-in. wide), count 2/28 (worsted).

Fabric B: Serge, 68 ends/in., 64 picks/in., weight 11·7 oz/yd (56-in. wide), count 2/42 worsted).

PAD-DYEING OF SLUBBING AND FABRIC

Both slubbing and narrow strips of fabric A could be readily padded with solutions of acid dyes in 90% formic acid, the pick-up of dye liquor being 100–150%. Maximum fixation of dye was reached after allowing the padded goods to stand for about 45 s, but generally 2 min were allowed. If 98–100%

TABLE IV

Degree of Fixation of Dyes at Two Concentration Levels

Dye	*Colour Index* No.	Degree of Fixation at 5 g/l.	Standard error	at 20 g/l.	Standard error
Coomassie Yellow R	Acid Yellow 42	93·8	3·5	69·4	7·8
Xylene Fast Yellow P	Acid Yellow 61	92·8	2·5	90·6	2·6
Xylene Fast Orange P	Acid Orange 43	106·0	3·7	88·5	4·0
Coomassie Red PG	Acid Red 85	97·3	2·4	74·2	5·5
Xylene Fast Red BL	—	100·9	6·1	90·7	9·2
Neolan Red REG	Acid Red 198	97·9	2·5	54·5	7·6
Carbolan Crimson 3B	Acid Red 139	105·2	4·2	78·3	6·6
Carbolan Brilliant Green 5G	Acid Green 28	103·1	4·2	71·0	4·5
Coomassie Ultra Sky SE	Acid Blue 112	82·4	2·9	47·4	3·3
Neolan Blue 2R	Acid Blue 154	93·2	1·4	64·4	5·6

formic acid was used as the dye solvent, only about 15 s were necessary for maximum dye fixation.

However, although maximum dye fixation was achieved within a minute or two, complete fixation of dye was not possible except at very low dye concentrations. Virtually complete fixation of many dyes was possible at concentrations of up to 2 g/l., but lower dye fixation was observed at higher dye concentrations. Table IV shows the degree of fixation of several dyes applied to slubbing at concentrations of 5 g/l. and 20 g/l. in 90% formic acid at room temperature. The slubbing was padded at the rate of 5 yd/min, and a fixation time of 3 min was allowed.

The following procedure was devised to minimise errors introduced by tailing and unlevelness, since examination of a cross-section of dyed slubbing generally showed that the outer fibres were the less heavily dyed. A 1-yd length of the padded slubbing was split lengthwise, one half being rinsed for 1 h in running water with frequent agitation, the other being dried in air. The samples were then conditioned at 20°C and 65% r.h. before dye analyses were carried out. Samples for analysis comprised three transverse sections taken from three equally spaced places along the length of slubbing, the dye being removed by continuous extraction with 80% pyridine in a Soxhlet apparatus, and estimated colorimetrically. The procedure was carried out four times for each dye concentration, the degree of fixation being represented by $100\,C_w/C_d$, where C_w is the dye concentration in the washed sample and C_d that in the unwashed sample. The means of these estimations are shown in Table IV. Only a few dyes give a satisfactory level of fixation at concentrations in the range 2–3%, although more afford satisfactory fixation at concentrations of up to 0·5%.

Although addition of either sodium chloride or sodium sulphate to dye solutions resulted in a marked reduction in the degree of fixation, the degree of fixation of purified dye samples was not appreciably higher than that of commercial dyes. Neither using 98–100% formic acid nor prolonging the fixation period improved the degree of fixation, while heating the padded goods at 100°C in formic acid vapour slightly improved the fixation in only a few cases. Comparison of the degree of fixation of Coomassie Red PG (C.I. Acid Red 85) with that of its free acid indicated that the use of the latter gave no advantage.

In addition to showing low fixation at higher concentrations, most dyes produce skittery dyeings when applied by a padding method, with consequent low visual colour yield. The skitteriness may be reduced for some level-dyeing acid dyes by prolonging the fixation time, the padded goods being kept in an atmosphere saturated with formic acid vapour. Incorporation of non-ionic, anionic, or cationic wetting agents in the dye liquor failed to reduce the skitteriness. Addition to the dye liquor of the thickening agent, gum guar, was also ineffective.

Medley and Andrews [6a] have studied the effect of the surface barrier (probably the epicuticle) which limits the rate of diffusion of dye molecules into wool fibres. They have subsequently found [6b] that addition of n-butyl alcohol to the dyebath markedly increases the rate of diffusion within the fibres, but affects the rate of diffusion through the surface barrier to a lesser extent, so that the overall effect is to make the surface barrier more significant than in dyeing from an aqueous dyebath. They have suggested [6c] that a similar phenomenon may occur in dyeing from a formic acid dyebath, and that any irregularities in the epicuticle would result in uneven dyeing along the fibres. However, we have found that skittery dyeings are still obtained on pad-dyeing slubbing which has been treated with a solution of potassium hydroxide in ethanol [7], a process known to destroy the epicuticle of wool fibres. Slubbing which has been acid-chlorinated also gives skittery dyeings when pad-dyed.

In a continuous process in which the dye has affinity for the goods in the dyebath, tailing or ending is always likely to occur. This is generally minimised by using a small-volume dyebath, the level being maintained by continuous addition of fresh dye liquor, and also by operating at the fastest practicable rate. In the equipment described, speeds of 10 yd/min could not be exceeded owing to inadequate wetting-out in a single dip. It has not been possible to dye sufficient lengths of slubbing to ensure that tailing does not take place, fifty yards being the greatest length dyed. However, it is believed that tailing would not present any problems so long as the usual precautions were taken.

Tightly woven fabrics and fabrics composed of high-twist yarns cannot be dyed satisfactorily by immersion in a solution of dye in formic acid, owing to inadequate penetration by the dye

liquor. Swelling of the surface fibres is believed to prevent further penetration of the fabric. The "penetration barrier" can be overcome if the dye liquor is padded into the fabric by means of squeeze rollers under high pressure. Good penetration of an open-weave fabric (A), a serge (B), and a gaberdine was possible by padding, although it was necessary to use 98–100% formic acid to ensure complete wetting of the serge and gaberdine fabrics. However, the dyeings all showed sandwich effects, the surface of the material being less heavily dyed than the interior, and also skitteriness. These effects have also been encountered in the application of dyes to wool fabrics using pad–steam processes [8].

Screen Printing

One of the main applications of the formic acid process would appear to be in the screen printing of woollen pieces [1]. A thickened solution of the dye in 90% formic acid is printed on to the fabric, which after 2–3 min is rinsed in water. Two of the most suitable thickening agents are Cellofas B (ICI) and gum guar (Karachi gum). Terylene screens blocked with certain rubber preparations have proved satisfactory. The process possesses advantages over the conventional method in that neither chlorination of the fabric nor steaming is necessary. Penetration is satisfactory, even with raised fabrics, and the process has been used for printing figure designs on blankets. The amount of formic acid used in this type of printing is small, and recovery is not necessary for economical application. However, the cost of printing to complete coverage would be considerable unless the acid could be recovered.

Solvent Recovery

In Australia the cost of dyeing tops is 1s. 0d.–2s. 0d./lb, depending on the depth of shade and the dyes used. When tops (as slubbing) are dyed by the formic acid pad-dyeing process, the pick-up of dye liquor is 120–150%. Using 90% formic acid (1s. 7d./lb in Australia) as the dye solvent, the cost of the acid alone will be 1s. 11d.–2s. 5d./lb of wool dyed. Obviously it will be necessary to recover most of the acid for re-use if the process is to become economically practicable. In some countries the cost of formic acid is less than half that in Australia, and it may be feasible to discard the used dye solvent.

Early attempts to recover the acid from pad-dyed slubbing made use of a blast of hot air to displace the acid. Although up to 90% could be removed with air at 100°C, the volume of air used was too great to permit the use of physical methods for subsequent recovery of the acid, and it was considered that chemical methods would be impracticable.

Efforts were then directed to the removal of formic acid from the pad-dyed slubbing by countercurrent washing with water, the acid concentration of the wash liquors being raised to as high a level as possible by these means before final concentration to the initial 90% by distillation. Unfortunately, formic acid forms a constant-boiling mixture containing approximately 77% acid, and concentration of a dilute acid solution to 90% necessitates azeotropic distillation, a more costly procedure than simple fractional distillation.

The countercurrent washing train used has been described above. Water was fed at a steady rate into the long bowl (7), passing through the train in the opposite direction to that of the padded slubbing, and finally flowing out from the first bowl (1). The concentration of the effluent acid was determined by titration, and that of the acid remaining on the wool as it emerged from each bowl along the train by the pyridine-extraction method [9].

Obviously, the acid concentration of the effluent varies with the rate of flow of water; the lower this rate, the higher the acid concentration of the effluent wash liquor. On the other hand, the lower the rate of flow of the water, the less efficient is the removal of acid from the slubbing. Much of the acid is removed in the first three bowls (1–3), the acid concentration on the wool falling only slowly as the slubbing passes through the subsequent bowls (4–7). Apparently, the acid held on the fibre surface is rapidly washed off, but acid held within the fibres diffuses only slowly into the wash liquors. This is borne out by the fact that a limiting concentration, about 10 g formic acid/100 g wool, was reached, irrespective of the rate of flow of the wash water. Possibly this amount could be reduced by extending bowl 7, so increasing the time of immersion of the wool in order that further diffusion of acid into the wash liquor could take place. By suitable adjustment of the rate of water flow, it was possible to obtain an effluent containing 40–44% formic acid, approximately 85% of the applied acid being so recovered. Experiments were also carried out using 75% formic acid as the dye solvent. In this case the wash liquors contained 18–25% acid, losses amounting to 16–17% of the total.

Wilson [10] has estimated that the cost of concentrating 25% formic acid to 75% would be approximately 2½d./lb, and concentration to 90%, approximately 5d./lb. Thus the cost of recovering 90% formic acid used in dyeing slubbing with a pick-up of 150% (assuming 15% loss on washing) would be 6¼d./lb of wool dyed, while replacement of the lost acid would cost a further 4¼d./lb, making a total of 11d. as the cost of formic acid required to dye one pound of wool. Using 75% acid as the dye solvent, the cost of recovering the acid and replacing the losses would amount to 7d./lb of wool dyed. A further cost would be that of heating the dye liquor to 40°C.

Even when the savings in steam and labour costs which the formic acid process afford are taken into consideration, the high cost of acid recovery and replacement of losses would probably preclude the economical application of the formic acid process using the method described.

Conclusions

Apart from the relatively high cost of recovery of the dye solvent, there are several other disadvantages suffered by this continuous-dyeing process. Of a large number of dyes tested, only relatively few yellow and red dyes afford a satisfactory degree of fixation at medium depths of shade. No green, blue or black dyes have been found which give a satisfactory degree of fixation. A further disadvantage is that the continuous-dyeing method gives rise to skitteriness with most dyes, only dyeings with yellow and orange acid dyes being satisfactory. Obviously, unless the problem of skitteriness can be overcome, and the range of suitable dyes extended, the process will not be a commercial proposition for continuous dyeing. On the other hand, the process possesses considerable advantages over the conventional method of screen-printing, and may be adopted for printing figure designs, and even printing to complete coverage, in countries where formic acid is obtainable more cheaply, and where the used acid could be discarded.

* * *

The author expresses his sincere thanks to Dr. B. S. Harrap, and Messrs. P. Maclellan, T. A. Pressley, and D. Y. Smith for many helpful discussions, to Mr. W. B. Hall for statistical analysis of the results, and to Dr. A. Datyner and Mr. D. Y. Smith for testing for fastness to rubbing and to light, respectively. Technical assistance by Messrs. D. T. Tucker and R. Munday is gratefully acknowledged.

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(*Received 25th October 1960*)

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FASTNESS TESTING

Committee on Consumer Protection

Recently the Society was asked by the Committee on Consumer Protection to comment on certain problems that had been raised before the Committee. A reply was sent on 14th September 1960, on behalf of Council, to Mr. S. W. T. Mitchelmore, Secretary of the Committee. Since this reply reviews the Society's work and policy in the field of fastness testing, it is considered to be of interest to all members. The letter is therefore reproduced below.

You have asked for the comments of this Society on the problems posed by the existence of a number of complaints relating to shrinking or to the fading or running of colours and on informative labelling.

This Society has been recognised since 1927 as the proper body to devise standard methods for testing the colour fastness of all textile materials and they are published by the Society under the title "Standard Methods for the Determination of the Colour Fastness of Textiles", the latest edition appearing in May 1960. The methods are developed by six technical sub-committees responsible to the Fastness Tests Co-ordinating Committee whose members are drawn from dye makers, dye users, research associations, textile manufacturers, retail organisations, etc. They are accepted by the British Standards Institution who first republish them in their Handbook No. 11—"Methods of Test for Textiles"—and later, when they have become fully established, as British Standards bearing *B.S.* numbers.

These methods enable the colour fastness of a textile material to be determined quantitatively and they are valid for all textile materials in any form, i.e. loose fibre, yarn or fabric, and coloured by any method, i.e. dyed, printed or coloured in the mass. They will also be equally valid for any new fibres which are made and for new colouring matters and methods of application.

Colouring matters vary widely in their fastness properties and it does not follow that a colouring matter which is of good fastness to light will have equally good fastness to washing or perspiration. Hence standard methods have had to be developed for no fewer than forty-three "agencies" which may change the colour of the material and/or which may cause it to stain adjacent white textile material. Most of these agencies occur during manufacture and will, therefore, not be of any interest to the Committee on Consumer Protection, but several occur after the textile material has been purchased by the consumer and thus will be of interest; these latter are as follows—

light, washing, perspiration, pressing, rubbing, water, sea water, swimming bath water, and dry cleaning.

Testing methods, which this Society believes to be both sound and satisfactory, exist for each of these agencies with the exception of dry cleaning, which is currently being studied by a sub-committee created specially for this purpose.

Full details of these testing methods which will enable any competent laboratory to carry out the tests are published by the Society and by B.S.I. in many cases, viz.—

Agency	Standard Methods for the Determination of the Colour Fastness of Textiles Second Edition 1960 Page No.	British Standard *B.S.*
Light	42	1006:1955
Washing	94	2680:1956
(several tests of	96	2684:1956
varying severity)	98	—
	100	—
	102	2685:1956
	104	—
Perspiration	67	—
Pressing	77	2676:1956
Rubbing	79	2677:1956
Water	107	2681:1956
Sea water	81	2682:1956
Swimming bath water	28	—

Each method provides a separate numerical rating for both the change in colour of the material and the staining of adjacent white textile materials, the rating 1 being given when the amount of change or staining is very great and 5 when there is no change in colour or no staining. The amount of change and staining corresponding to each of the five fastness grades is illustrated by Grey Scales which are available from the Society's offices. In the case of light fastness, the method of assessment is somewhat different for rather complicated technical reasons; here Grey Scales are not used for assessment and eight grades are recognised, 1 again being the most fugitive, but 8 is the most resistant to light.

Whilst these methods are believed to be both sound and satisfactory, the Fastness Tests Co-ordinating Committee has recently found it necessary to append the following warning footnote to all its washing tests—

> This method of testing is established as a guide to the determination of the fastness to washing of coloured textiles. It takes no account of any effect of fluorescent brightening agents or other reactive chemicals which may be present in commercial soaps and detergents or from other sources.

This was necessary as the Committee had found it impossible to devise a sound and satisfactory method to cover the soap and detergent preparations on the market today which vary so markedly in their composition; in particular, the effect of fluorescent brightening agents often gives the impression that the colour of the fabric has changed whereas the agent has actually masked it—it was quite impossible to devise a standard test which would cover such effects.

It must be appreciated, however, that even if coloured textiles are subjected to these tests this cannot, in itself, ensure freedom from complaint nor even reduce the incidence of complaints. Some body has to decide that for each end-use a certain minimum rating must be achieved and this rating will vary according to the end-use; it is obvious, for example, that curtain material should have a higher light fastness than material intended for dresses or tablecloths, and engineers' overalls a higher washing fastness than woollen knitwear.

The selection of these minimum ratings constitutes the establishment of standards of performance which is the province of the British Standards Institution, who have already issued a few performance standards concerned with the colour fastness of textiles, e.g.—

*B.S.*2543:1954— Woven Upholstery Fabrics

*B.S.*2802:1956— Cotton Velveteens for Loose Cushions

*B.S.*3153:1959— Cotton Furnishing Fabrics

The minimum ratings in *B.S.*3153 show how the Society's methods are utilised by B.S.I. in establishing performance standards and, incidentally, the complexity of some performance standards—

Light fastness— When tested by *B.S.*1006 (devised by this Society) the minimum light fastness must be 4, 5, 6 or 7 according to the depth of shade and the type of fabric (B.S.I. requirement).

Washing fastness— When tested by the Mechanical Washing B test (devised by this Society) the washing fastness must be not less than grade 4 for change in colour and grade 3–4 for staining of adjacent cloth (B.S.I. requirement).

Being a professional society concerned with the science and technology of colouring matters and their application, we do not participate in the establishment of performance standards other than in providing the testing methods. We cannot, therefore, comment on whether these British Standards of performance can be accepted as sound and satisfactory nor can we comment on possible statutory provisions.

With regard to informative labelling we think that the above evidence suggests that a label which expressed colour fastness to light, washing, and other agencies would be meaningless to the consumer, as the whole subject is much too complicated technically. Reference to a satisfactory British Standard of performance is a different matter, but there are relatively very few of these at the moment. A label giving information on washing instructions and use in the case of fabrics sold by the yard would be more helpful.

Presentation of Medals and Diplomas

A special ceremony for the presentation of medals and diplomas was held on Wednesday 1st March 1961 at the Victoria Hotel, Bradford. About 170 members of the Society and guests assembled for afternoon tea, after which the ceremony took place.

Mr. F. M. Stevenson, J.P., M.Sc., F.S.D.C., President of the Society, opening the proceedings, said that the event was in the nature of an experiment. Up to that afternoon the Society had hoped for success; now it was obvious that success was assured. He extended a special welcome to the ladies, without whom the world would indeed be drab, to representatives from industry, who had sacrificed valuable time to lend their support, and to those from the academic world, who played an important role in the Society's life. This was the first occasion on which such a function had been held, and the people taking part were creating history. In the past we had, perhaps, tended to hurry through the presentation of medals at the Annual Dinner. But these medals, and the Diplomas of Fellowship and Associateship, were awards of great importance and value. The Society was an important influence in the scientific and industrial life of the country. He hoped that the award winners would realise how much the Society appreciated their presence on this occasion.

Mr. L. Morton Wood, Chairman of the Medals Committee and Honorary Secretary of the Society, said it was his happy duty to present the Medallists. Medals were hard to win; often the Medals Committee would like to present more medals, but it was necessary to restrict awards to those of outstanding merit. He then introduced the Medallists as follows—

Gold Medal

William Alfred Edwards, F.T.I., C.G.I.A., F.S.D.C., joined the Society in 1911, was awarded the Silver Medal in 1936, and was made a Fellow in 1954. He was a founder member of the Midlands Section and of the recently formed Midlands Junior branch. He had served on Council and on many committees, but matters of education were uppermost in his mind and he had been associated with the Diplomas Committee since 1952.

William Penn, F.T.I., F.S.D.C., joined the Society in 1928, and was elected a Fellow in 1954. He had been a member of Council, in one capacity or another, for several periods since 1946. He was one of the pioneers of the Midlands Section. Education was one of his particular interests, and he had served on Diplomas Committee since 1951. His judgment and guidance had proved invaluable to Finance and General Purposes Committee, Examinations Board, and several other committees.

Silver Medal

Rowlay Grice joined the Society in 1911, was a Vice-President from 1940 to 1948, and was very active in the formation of the Bradford Junior Branch. He had been a prodigious worker for the West Riding Section. Since 1929 he had sat on Publications Committee and in recent years had acted as scrutineer of the ballot for Vice-Presidents and members of Council.

Edgar Irvine Noble, M.Sc., A.R.A.C.I., F.S.D.C., joined the Society in 1922 and became a Fellow in 1955. He was an enthusiastic worker for the Society in Australasia, and had done much to increase its membership there. On two occasions he had been President of the Society of Dyers and Colourists of Australia.
(The President read a telegram of greetings from Mr. Noble, who was unable to be present. Mr. G. G. Bradshaw received the medal on Mr. Noble's behalf.)

Cyril Beynon Stevens, B.Sc., Ph.D., F.S.D.C., became a member in 1933 and a Fellow in 1954. He served on Council, Publications Committee, Diplomas Committee, Examinations Board, and other committees, and was a past Chairman of the Leeds Junior Branch. He had published many papers on dyeing topics, especially in the *Journal*.

Bronze Medal

Frank Harrison, A.M.C.T., joined the Society in 1947 and since then had served continuously as the Honorary Treasurer of the London Section.

Research Medal of the Worshipful Company of Dyers of the City of London

Mr. Wood announced that this medal, which was awarded on the recommendation of a committee of the Society, had been awarded by the Court of the Company to Dr. Jacques Wegmann, who would receive the medal at a Dinner of the Company in London.

Mr. Stevenson next called on Mr. Clifford Paine, B.Sc., F.R.I.C., F.S.D.C., Chairman of the Diplomas Committee and a Past President of the Society, to introduce the Fellows and Associates who were to receive Diplomas.

Mr. C. Paine, in his introductory remarks, hoped that this function would grow in importance and value over the years, both to the Society and to the Fellows and Associates. It was appropriate to recall that the Society was founded over 75 years ago to promote scientific and technical knowledge among textile colourists. This was less than 30 years after the discovery of the first synthetic dye, a British achievement. The founders of the Society were very anxious about subsequent developments and felt that the British industry was losing

WILLIAM ALFRED EDWARDS
F.T.I., C.G.I.A., F.S.D.C.

Gold Medal for exceptional services to the Society

WILLIAM PENN
F.T.I., F.S.D.C.

Gold Medal for exceptional services to the Society and to the tinctorial and allied industries

JACQUES WEGMANN
Ph.D.

Worshipful Company of Dyers' Research Medal for 1959–60 as author of a paper on *Effect of Structure on the Change in Colour of Vat Dyes on Soaping*

ROWLAY GRICE

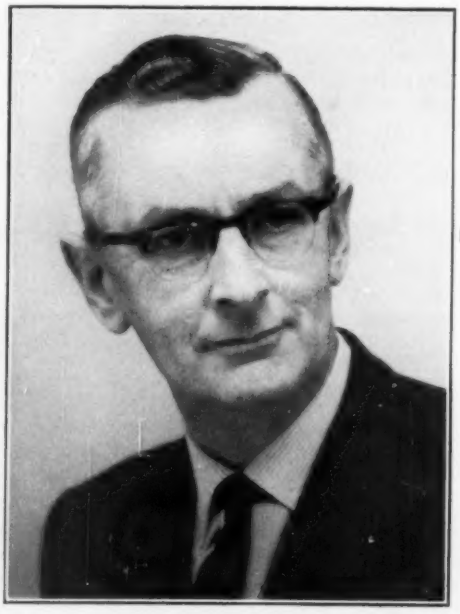
Silver Medal for valuable services to the Society

EDGAR IRVINE NOBLE
M.Sc., A.R.A.C.I., F.S.D.C.

Silver Medal for valuable services to the Society, especially in Australasia

CYRIL BEYNON STEVENS
B.Sc., Ph.D., F.S.D.C.

Silver Medal for valuable services to the Society



FRANK HARRISON
A.M.C.T.

Bronze Medal for valuable services to the Society since 1947 as Honorary Treasurer of the London Section

ground to its Continental competitors. Before 1914 they were, unfortunately, crying in the wilderness, but the advent of the war showed how ill-equipped the country was in the science and technology of the dyestuffs industry. Since then the Society and all it stood for had increased in importance in the national life. The Society of today had an enormously widened scope; it had international contacts, and was concerned with the coloration of all types of substrate.

The Associateship Examination was one way in which the Society endeavoured to maintain standards. Associates could be proud of their achievement, but should look upon their Associateship not only as desirable evidence of a standard of skill and knowledge, but as a bond between themselves and the Society. The Associate of today was the potential Fellow of tomorrow. The Fellows had the right to feel that they were being recognised as having made mature contributions to science and technology or to leadership in industry.

The best contribution that Fellows and Associates, and indeed all members, could make would be to take an active part in the work and efforts of the Society, for only so could its objects be achieved. He and his colleagues on the Diplomas Committee felt their work to be important and worthwhile—and the very onerous work of the Examinations Board should not be overlooked. Council had recently, on the recommendation of the Diplomas Committee, set up an Education Committee to consider what standards would be required, say 10–15 years hence, so that the Society was planning well for the future.

It was his privilege to present the newly elected Fellows and Associates to receive their Diplomas from the President.

Mr. J. W. Nicholls, F.C.I.S., General Secretary of the Society, read out the names of Fellows and Associates, who then received their Diplomas. A few were unable to be present, and it was announced that arrangements would be made for them to receive the Diplomas later.

FELLOW

James Harry France Hilton, A.M.C.T., A.T.I.
Walter Hargreaves Hindle, F.T.I.
Geoffrey Pullan Pearson, M.Sc.

ASSOCIATE

Kenneth Alan Bagley
Anthony John Baker
Brian Bennett
James Ernest Blavins
Bernard Charnley
Zouhair S. Debs
Joseph Devey
Abd-el-Khalik El-Awad
Derek Alan Ferguson
Stanley Finlay
David John Garner
Ronald Steele Harding
Frederick Thomas Higgins
Wladyslaw Filip Luczynski
Alan Henry McKeand
Anthony Charles Pitman
James Samuel Pittillo
Robert Sidlow
Julius Victor Szolin
James Kenmure Mitchell Weir
John Malcolm Wilkinson
Peter Wood

Mr. F. M. Stevenson called upon Mr. John Boulton, M.Sc.Tech., F.R.I.C., F.T.I., F.S.D.C., President of the International Federation of Associations of Textile Chemists and Colourists, and Immediate Past-President of the Society, to close the proceedings.

Mr. Boulton said that it was his privilege, before closing the happy proceedings, to thank all who had made the occasion possible. He would like to add his own congratulations to the Medallists, and to remind those new Associates from the Midlands that if they aspired to emulate the achievements of the Gold Medallists or of the President they would need to do rather more than just come from the same part of the country. The standard of the Associateship was very high indeed. In fact, the scheme had become so successful that it was an object of envy and admiration to many countries overseas.

Mr. Boulton thanked Mr. Paine and the members of Diplomas Committee and the Examinations Board for the work they had done in developing the whole educational scheme, and the Staff, on whom a great burden fell, and in whom the Society had great confidence. Finally, after thanking Mr. Morton Wood for introducing the Medallists and the President for the gracious way in which he had conducted the Proceedings, Mr. Boulton declared the presentation ceremony closed.

Notes

Council Meeting—4th January 1961

Among the matters discussed at the meeting of Council on 4th January were—

PRESIDENT-ELECT—It was unanimously resolved that Council nominate Vice-President Richard Charles Oakley for election at the next Annual General Meeting to be President-elect as from 28th April 1961, and to become President in April 1962.

MEMBERSHIP—41 Ordinary Members and 11 Junior Members were elected.

FORMATION OF JUNIOR BRANCHES—An application was received from the Midlands Section Committee for authority to form two junior branches, to be known as "Midlands Section Leicester Junior Branch" and "Midlands Section Nottingham Junior Branch". The application was supported by the authorities of the Leicester and Nottingham Technical Colleges. Council authorised the formation of the two junior branches.

DYERS' COMPANY RESEARCH MEDAL—It was resolved to ask the Clerk of the Court of the Worshipful Company of Dyers to convey to the Court the appreciation and thanks of Council for the award of the 1959–60 Research Medal to Dr. Jacques Wegmann, and for the offer of a medal for the period July 1960–July 1961.

I.F.A.T.C.C.—Council considered a report on the proceedings of a meeting of the Special Sub-committee of Delegates of the I.F.A.T.C.C. held in Paris on 9th December 1960. It was resolved that the International Relations Committee should examine the Society's further participation in the moves being made to widen the scope of the Federation and that the Diplomas Committee should prepare a report on the Society's present educational schemes which might be helpful to the Sub-committee of I.F.A.T.C.C. in its deliberations.

LAMPS FOR COLOUR MATCHING—It was agreed to circulate a questionnaire to interested members of the Society, and to the trade organisations interested in the use of daylight lamps, in order to ascertain their views on the problems of constructing a suitable colour-matching lamp.

PHOTOCHEMISTRY—Attention was drawn to the fact that much work had been done in the field of photochemistry since the symposium on this topic, and it was agreed that the Society's next symposium, which was due to be held in 1963, should be devoted to aspects of photochemistry, unless it was found that this subject could be dealt with at an earlier conference of the Society.

PHOTODEGRADATION—It was reported that as a result of an approach made by the Society to the Textile Institute, a Technical Committee had been set up to devise standard methods of test for the assessment of degradation of textiles which had been exposed to the atmosphere and to light.

BRITISH UNITED PROVIDENT ASSOCIATION—A report was received that 100 persons drawn from families of members of the Society were now participating in the Society's scheme for private treatment in hospital.

A.S.D.C. Examination 1961

Owing to circumstances beyond the Society's control it has been necessary to change the dates on which the 1961 Associateship examination will take place. It will now be held on Thursday, Friday, and Saturday, 25–27th May 1961.

Commonwealth Technical Training Week

The Commonwealth Technical Training Week, 29th May–4th June in the U.K., is designed to stress the importance of training for all types of employment and is planned to arouse an awareness of the responsibility of the whole community to young people entering employment. Information about the activities arranged may be obtained from The Secretary, Commonwealth Technical Training Week, 76 Portland Place, London, W.1.

Mercer Lecture

The Mercer Lecture, which was to have been given on 17th May 1961 at the Midland Hotel, Manchester, has been postponed. The new date will be announced as soon as possible.

Maxwell Colour Centenary

On 17th May 1861 James Clark Maxwell gave his famous demonstration of trichromatic colour reproduction at the Royal Institution in London. To mark the centenary of this event, The Colour Group, in collaboration with The Institute of Physics and The Physical Society, and The Inter-Society Color Council of the U.S.A., has arranged a conference, to be held on 16th–18th May 1961 at the Imperial College of Science and Technology, South Kensington, London, S.W.7. In addition, a centenary discourse will be given at the Royal Institution on the evening of 17th May by Dr. D. A. Spencer, the title being "A hundred years of colour photography".

The subjects for discussion on the three days will be Trichromatic Principles, Colour Reproduction, and Colour Appearance. Each session will be opened by a short review of the current position of the subject concerned, followed by papers by one or two well-known workers in the field; time will then be allowed for contributions by other conference members and for discussion. Extended abstracts of the invited papers will be available.

Application forms are obtainable from, and should be returned (not later than 31st March) to

The Institute of Physics and The Physical Society, 1 Lowther Gardens, Prince Consort Road, London, S.W.7.

Chemical Society Library

The Library of the Chemical Society will be closed for the Easter holidays from 1 p.m. on Thursday, 30th March until 9.30 a.m. on Wednesday, 5th April.

Explosion in Analytical Laboratory

In a letter published in *Chem. & Ind.* No. 5 (4 Feb. 1961) 140, J. Thomson describes an explosion that occurred when a red azo pigment, C.I. Pigment Red 38, was wet-oxidised by the method of Smith & Diehl (*Talanta*, 4 (1960) 185–193), described by the authors as hazard-free. The technique was being considered for use in the analysis of trace metals in pigments, and a sample was being examined, using a slight modification of the Bethge apparatus. The sample (5 g) was added to the 250-ml. round-bottomed flask, with 60% perchloric acid (15 ml), 50/50 (wt./wt.) paraperiodic acid (3 g), and sodium metavanadate (1 mg). Slight heat was applied to promote reaction. After 2–3 min, heat was removed and the reaction proceeded as expected. Reaction slowed down, so slight heat was again applied; boiling recommenced. Suddenly the mixture in the flask began to darken considerably; heat was removed and a safety shield put in place. Flashes increased in number and proportions, and every 5 s or so a more violent flash was observed. About 1 min from start of darkening the flask shattered completely, but the reflux system and condenser were left intact. Because of the warning signs and the time lag no injuries were sustained by the operator.

Textile Institute Convocation

At the Jubilee Year Convocation of the Textile Institute, held in Manchester Town Hall on 16th November 1960, Professor C. S. Whewell, Ph.D., F.R.I.C., F.T.I., F.S.D.C., was awarded the Warner Memorial Medal, in recognition of outstanding work in science and technology, particularly in relation to work published in the *Journal of the Textile Institute*. Mr. N. G. McCulloch, C.B.E., F.S.D.C., was made a Companion of the Textile Institute. The newly established Jubilee Award was presented to "all those members (both past and present) of the staff of the Tootal Broadhurst Lee Company Limited who contributed to the research work which resulted in their crease-resist process".

Death of Member

We regret to report the death of Mr. L. I. Barford.

Meetings of Council and Committees

February

Finance and General Purposes— 14th
Education— 15th
Colour Index Editorial Board— 20th
Publications— 21st
International Relations Subcommittee— 22nd
Fastness Tests Co-ordinating— 28th

OBITUARY NOTICE

Norman Forbes Buchanan McLean

Norman McLean, who died on 8th May 1960 at the age of 56, was a member of the Society for some thirty years.

After studying at Loughborough College, he obtained an external B.Sc. degree in chemistry from London University in 1923. After several years with Joseph Lucas Ltd. in Birmingham and subsequently as dyehouse manager of Cheadle Textiles Ltd., interest in the dyeing of rayon filament yarn led him to establish, in 1937, the firm of Bank Yarns Ltd., in Cheadle, where rayon yarn was dyed by a new process which he had invented. War-time conditions compelled him to close the business, and in 1942 he joined the staff of the Royal Ordnance Factory at Swynnerton. From there he went, in 1946, to B.I.P. Chemicals Ltd., joining the Textile Resin Section of the Research Department, where he remained until his death.

McLean was a popular member of the staff and was particularly successful in training junior staff. His reputation as a textile chemist was high, and his name appeared on several patents relating to the application of amino resins to textiles.

New Books and Publications

Introduction to Colloid Chemistry

By Karol J. Mysels. Pp. xvi + 475. New York and London: Interscience Publishers. 1959. Price, 75s. 0d.

Fashions in science bear no relation to the importance of the subject. They are as capricious as fashions in women's clothes, and at the moment, colloid chemistry is unfashionable. This book is a welcome reminder of its far-reaching importance in modern life. The subject is dealt with in an extensive though elementary way. In places, the author's pursuit of clarity leads to repetition, but this is a fault that will be regarded as an asset by many students.

The chapter headings are: Why colloid chemistry?; Some structural elements; Sedimentation rate; Some aspects of flocculation; Diffusion and Brownian motion; Colligative properties; Sedimentation equilibrium; Sorption; Some applications of sorption; Fluctuations and

rubber elasticity; Preparation of colloids; Descriptive rheology; Structural rheology; Electroneutrality; Double layer; Electrokinetics; Some charge effects in colloids; Optical phenomena: I Refraction and schlieren, II Interference, III Scattering; Index. Each chapter is followed by a small section of problems of varying degrees of difficulty.

The book is well-produced, the diagrams are clear, and typographical errors are few. It is the author's intention that his work should be useful to university students and to industrial chemists, and I am sure he will succeed. It will be of undoubted value to those unfortunates of the second group who have been brought up in schools where colloid chemistry is a neglected field.

E. V. Truter

Oberflächenaktive Anlagerungsprodukte des Äthylenoxyds, ihre Herstellung, Eigenschaften und Anwendung

By N. Schönfeldt. Pp. xvi + 452. Stuttgart: Wissenschaftliche Verlagsgesellschaft m.b.H. 1959. Price, DM 68.00.

Since they were first prepared about 30 years ago, ethylene oxide condensates have become of importance in many industries, and a profusion of scientific and technological data exists dealing with their production, properties and applications. Dr. Schönfeldt's book, which is an attempt to collect and classify this material, contains 950 references to the original literature, in addition to numerous patent references, and deals with the manufacture, physical and chemical properties, applications and analysis of the condensates. An appendix is included which gives a complete list of commercial ethylene oxide adducts, together with their chemical compositions and applications.

The first two chapters deal with the manufacture of ethylene oxide and the condensates, and then follows a scheme for classifying different types of adducts. Chapter III covers mainly the solution properties, with short paragraphs on the biological and corrosion-inhibiting properties. Unfortunately, much of the fundamental work reported was carried out on commercial materials which would not only contain a distribution of molecular weights but might also contain impurities, e.g. unchanged fatty alcohols or phenols. Little systematic work has so far been done in this field, and as yet there are no results of physical measurements which would compare with the very accurate data which exist on anionic and cationic synthetic detergents. Nevertheless, the accounts of structure, viscosity, surface activity, etc., make interesting reading, although at times more comment by the author might have been helpful. It is stated, for instance (p. 98), that the viscosity of solutions of a given ethylene oxide–alkyl phenol condensate first decreases with rise in temperature, passes through a minimum (near 40°C) and then increases up to the cloud point, above which it rapidly falls. In the reviewer's opinion it is not sufficient to explain such strange behaviour by a brief mention that it might be due to solvation. In an otherwise complete section on structure it is surprising to find no mention of the use of the electron microscope (Rösch) in the investigation of the solid and micellar states.

Chapter IV gives an account of the applications of the condensates in 10 industries as diverse as laundering, textiles, pharmaceuticals, and metals. This is probably the most valuable part of the book, and it includes sections on generally little-known uses of the condensates, for instance as latex stabilisers, cement additives and antioxidants. Chapter V gives a short account of sulphated condensates, and Chapter VI deals with their analysis. Unfortunately, only 4½ pages are devoted to their quantitative estimation, and no single method is described in sufficient detail to obviate the need for consulting the original literature. Since much of the work in this field has been published in journals not readily accessible to most chemists, this lack of detail is a decided disadvantage.

In spite of these minor criticisms, the book will prove useful to those carrying out research on surface activity, and indispensable to technologists in industries where ethylene oxide condensates are already used or where their applications are yet to be developed.

F. Franks

Textile Terms and Definitions

Pp. 167. Manchester: The Textile Institute. 1960. 4th Edition. Price, 17s. 6d.

The first edition of this glossary was published in September 1954 and contained some 500 terms. The aim, as expressed in the Foreword to the first edition, was to provide definitions, as simple and as terse as possible, yet at the same time unambiguous, which would be of value to the student, the qualified technician or technologist, and the industrialist. The success of the venture is reflected in the publication of three further editions within six years, the glossary now containing about 1700 terms.

The fourth edition contains the revised substance of the first three, together with new terms relating to defects and fabric faults, and cloth assessment in terms of cover factors and cloth geometry. Definitions that have been discussed with the Terms and Definitions Committee of the S.D.C. are indicated by an obelisk. M.T.

Moisture in Textiles

Edited by J. W. S. Hearle and R. H. Peters. Pp. ix + 203. Manchester & London: The Textile Institute and Butterworths Scientific Publications. 1960. Price, 40s. 0d.

This book is based on a series of lectures organised by Dr. J. M. Preston at the Manchester College of Science and Technology. The lectures as delivered provided an excellent, all-round treatment of the subject, and the same may be said of the book which they inspired. The contributors to the various specialist chapters are or have been practising experts in some aspect of moisture relations of textiles, and include such well-known names as Urquhart, Meredith, Crank, and King.

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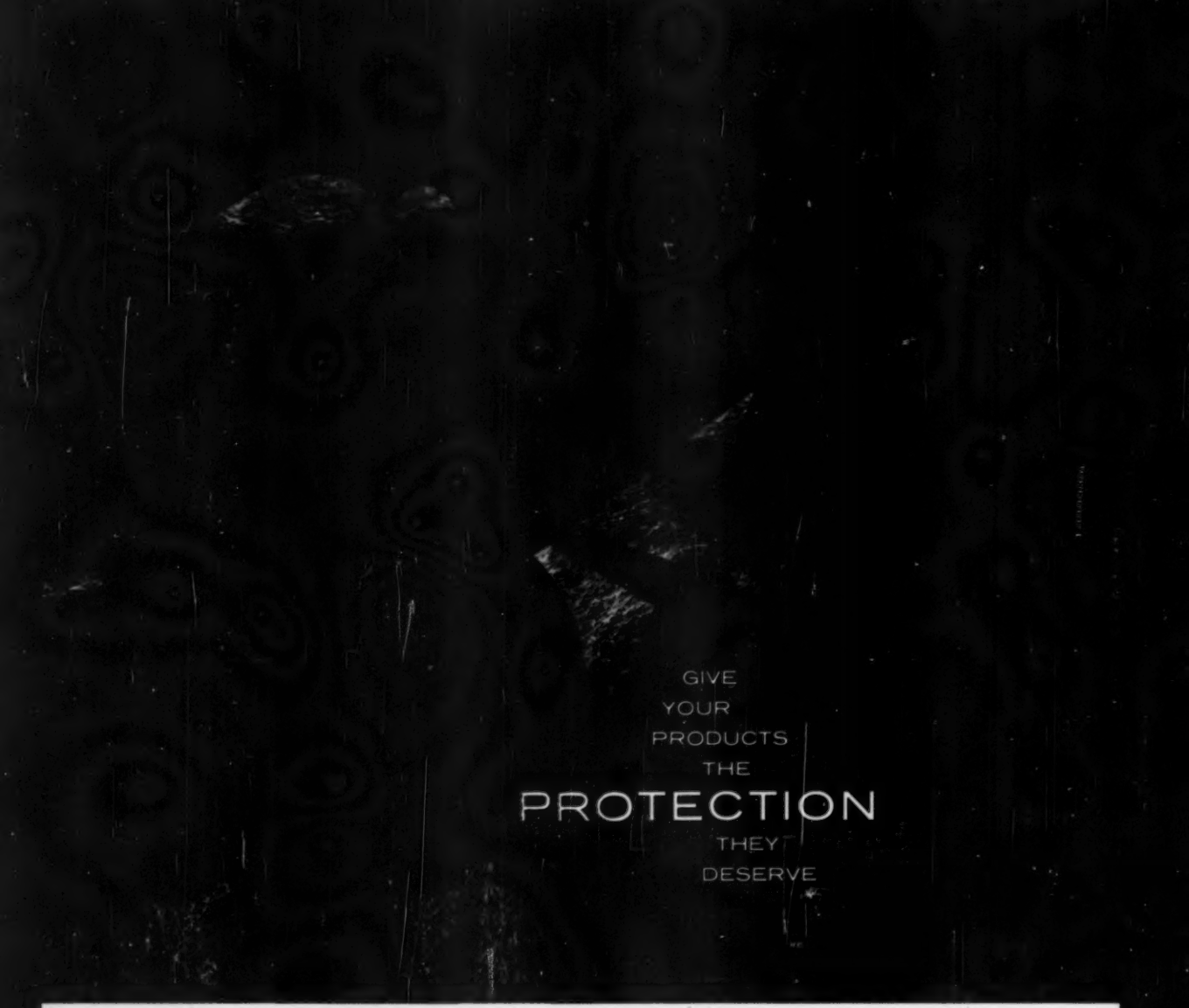

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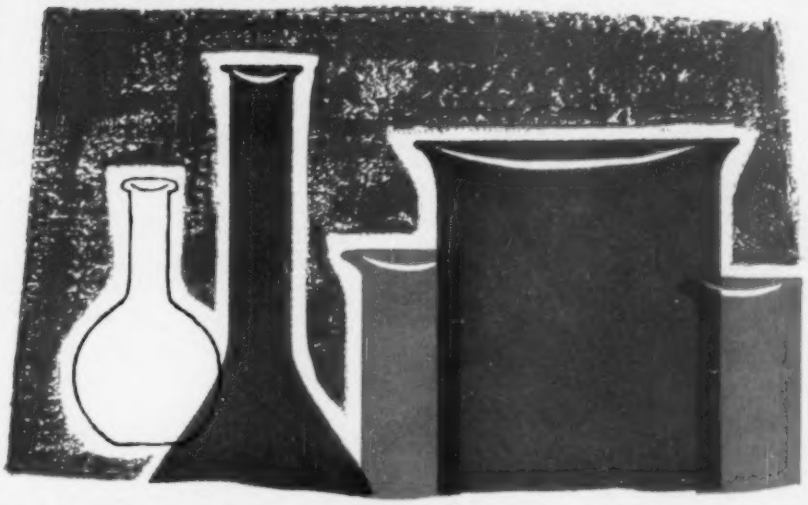

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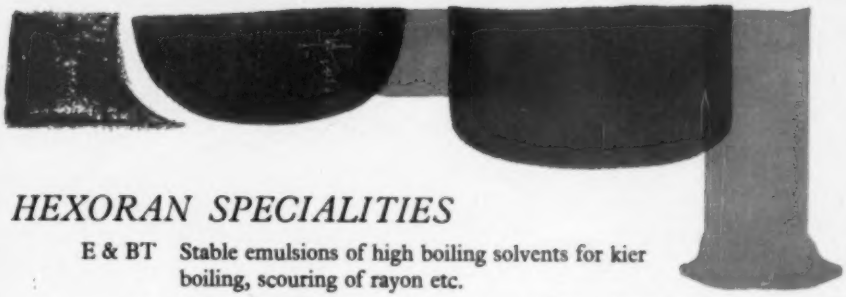

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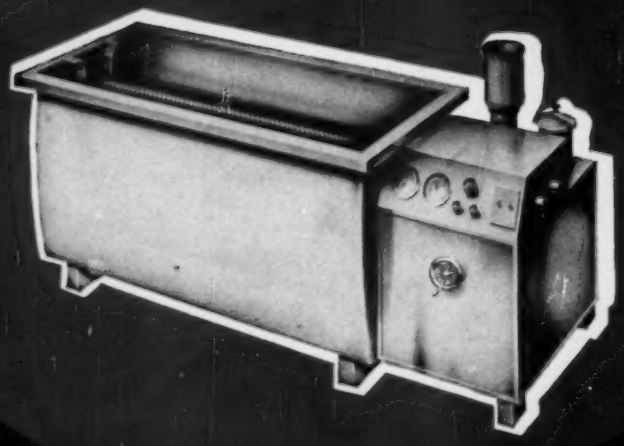

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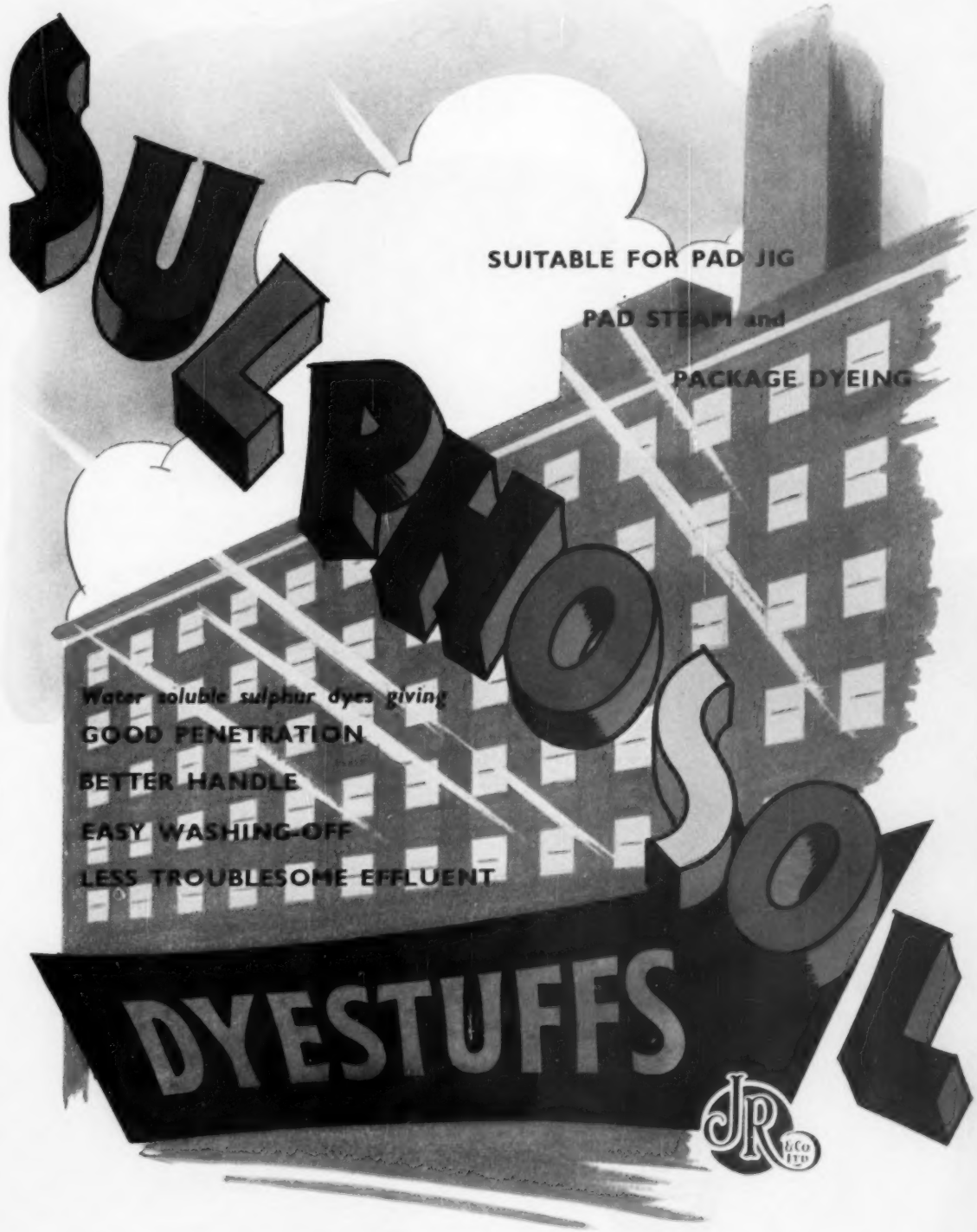

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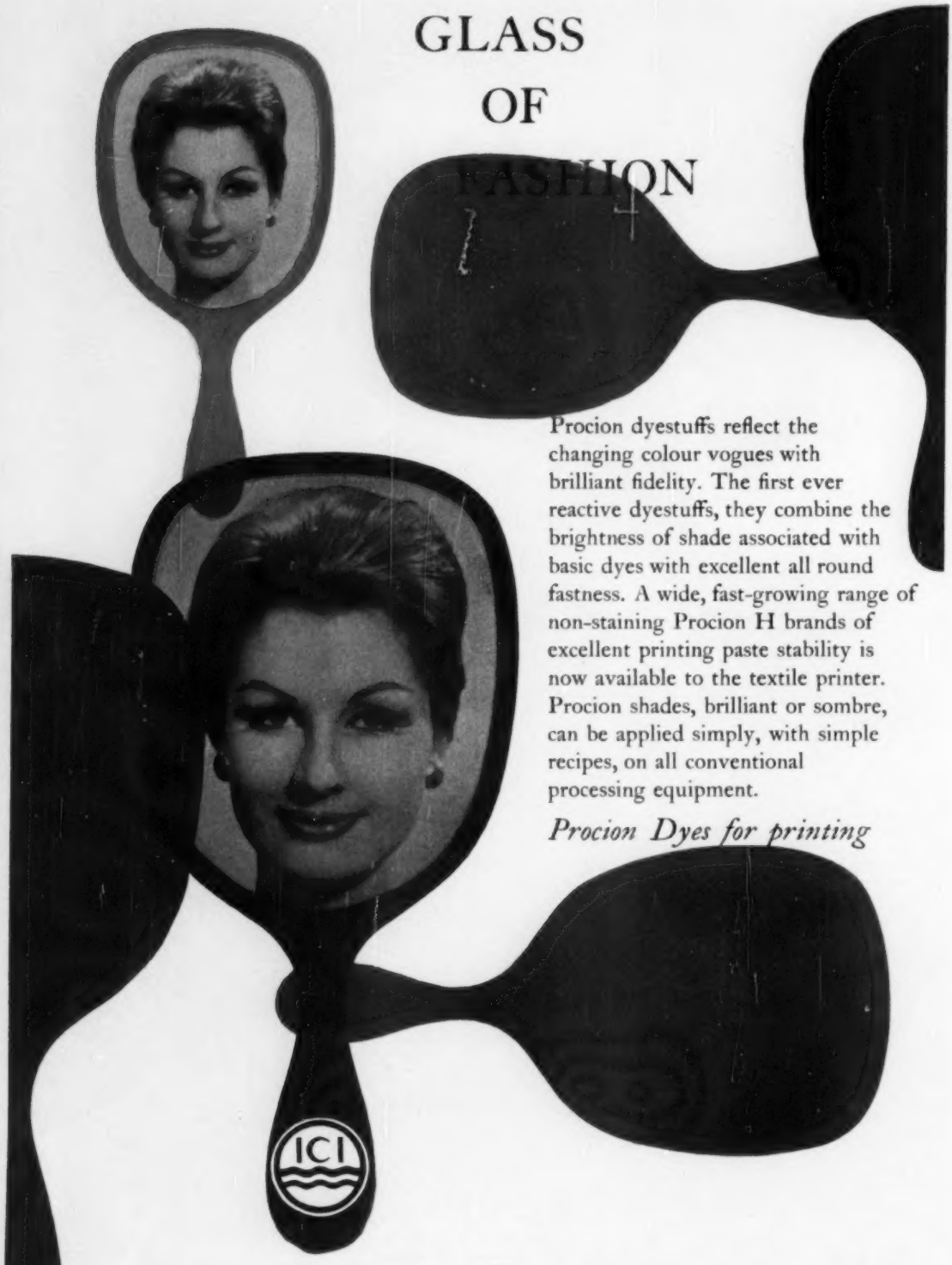

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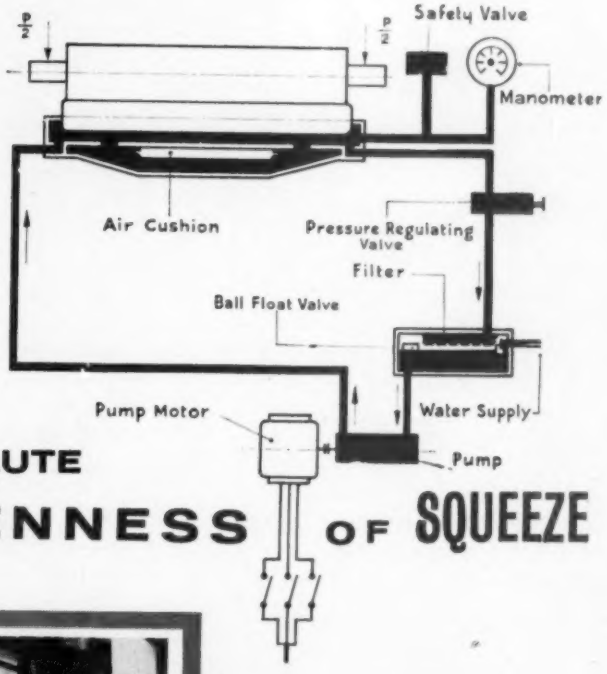

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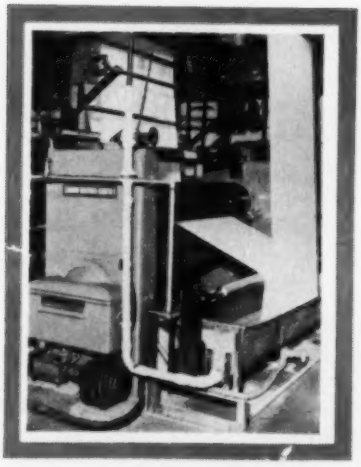

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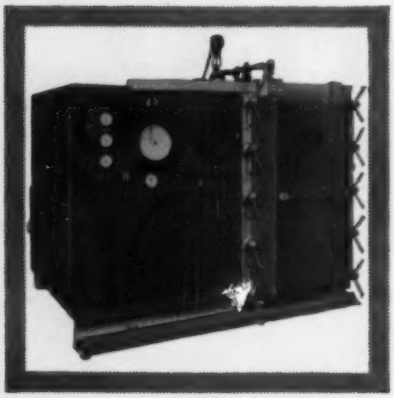

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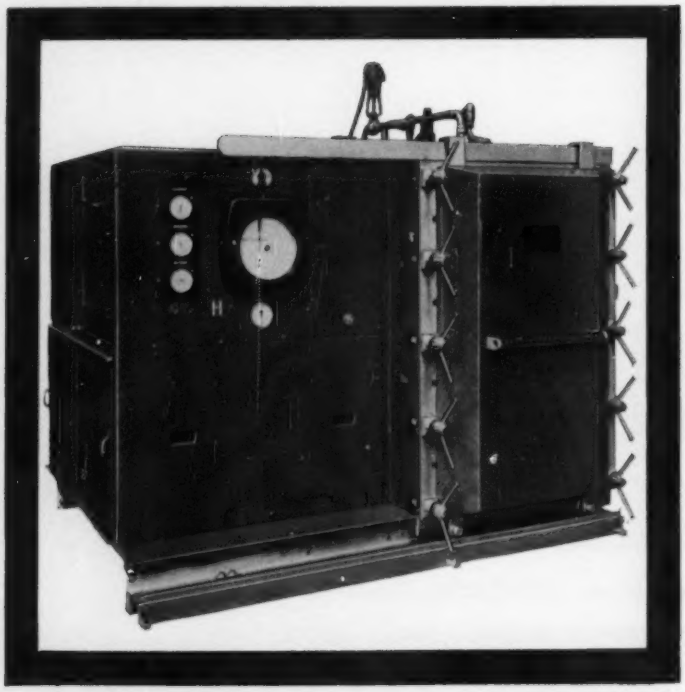

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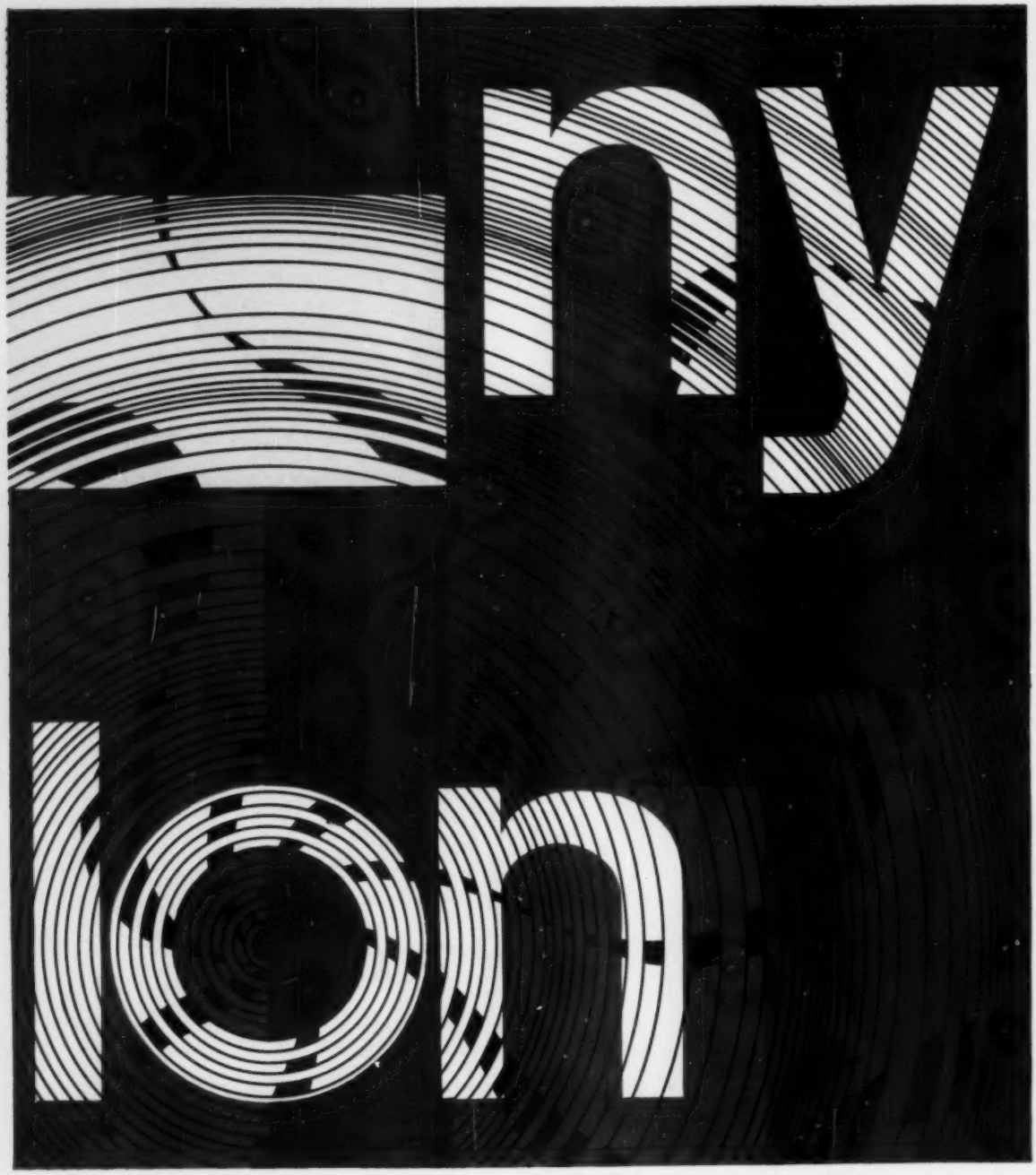

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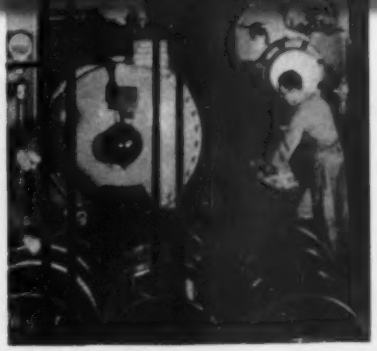

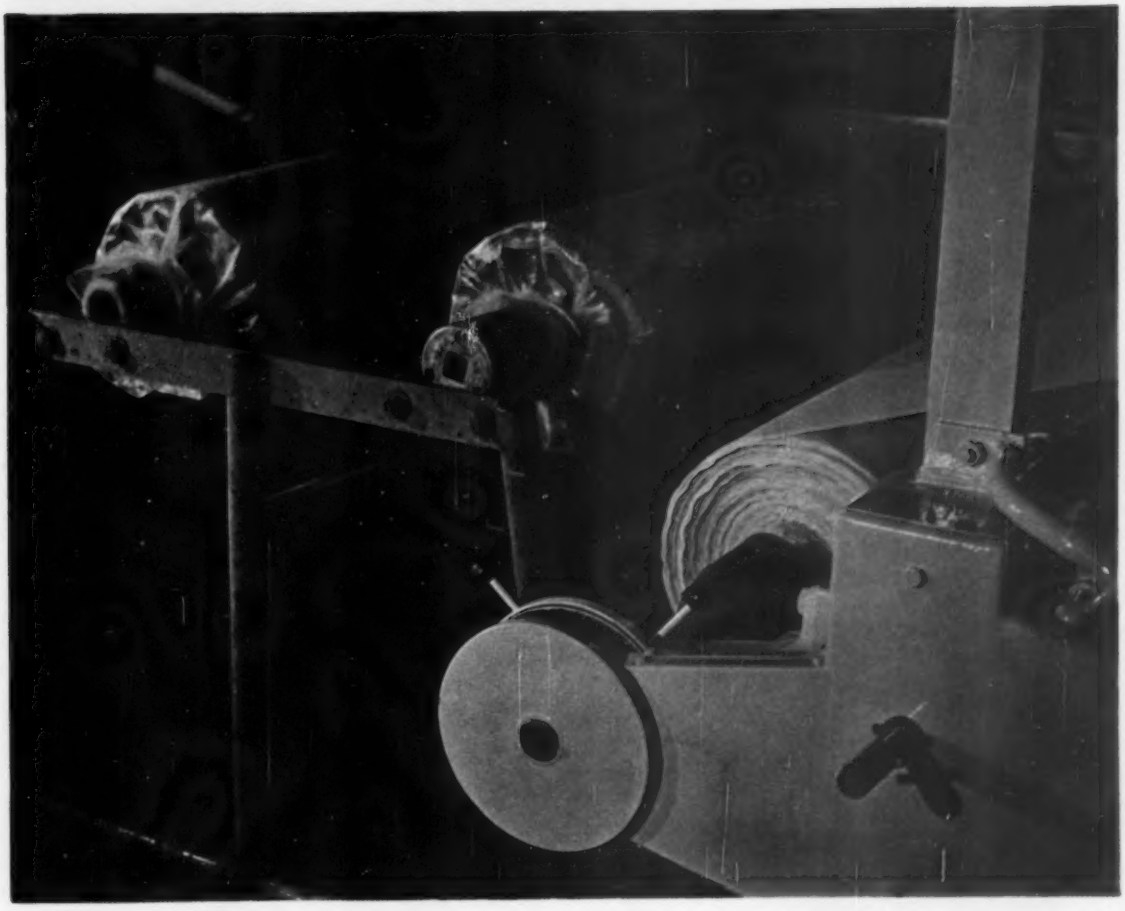

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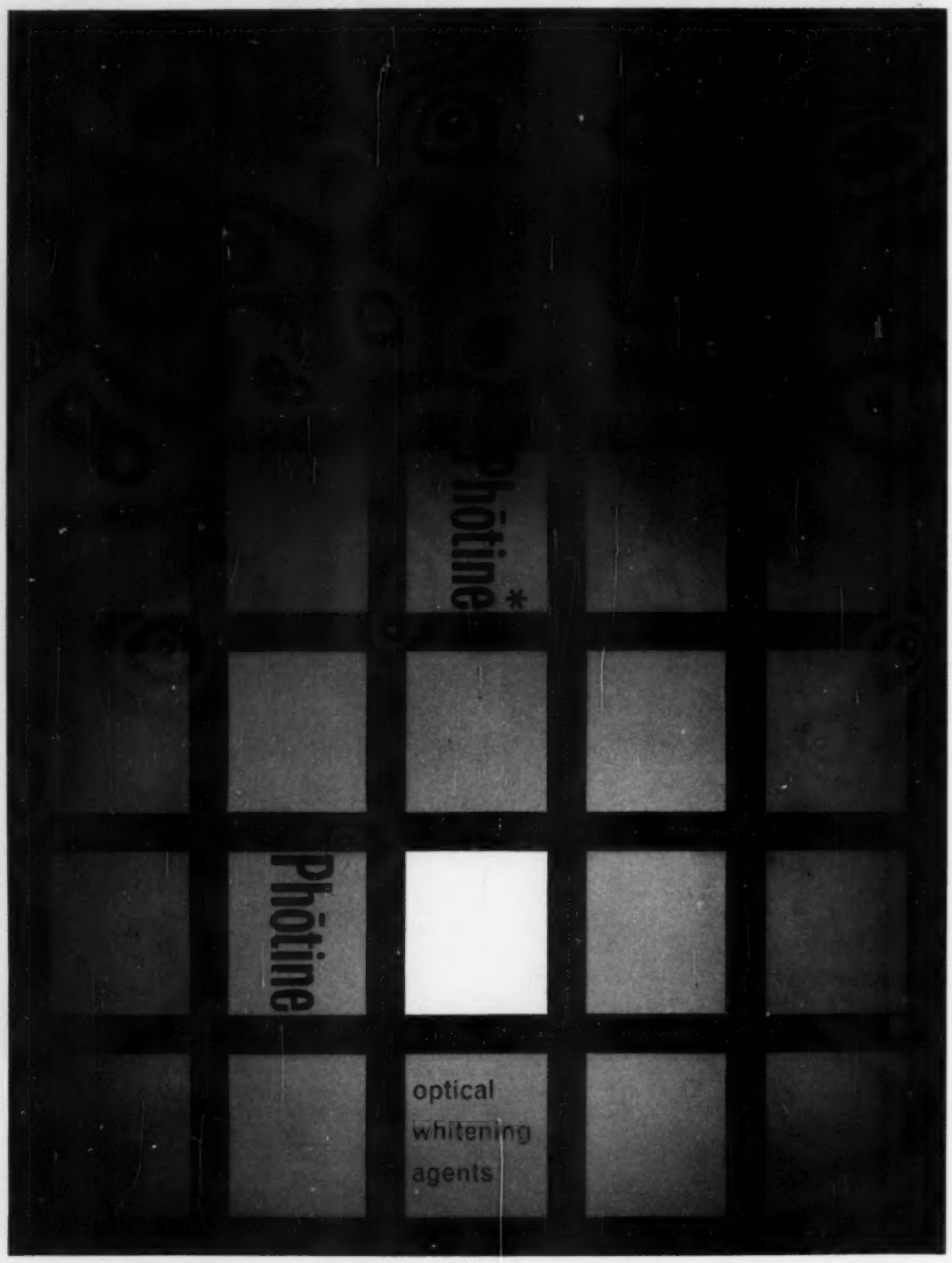

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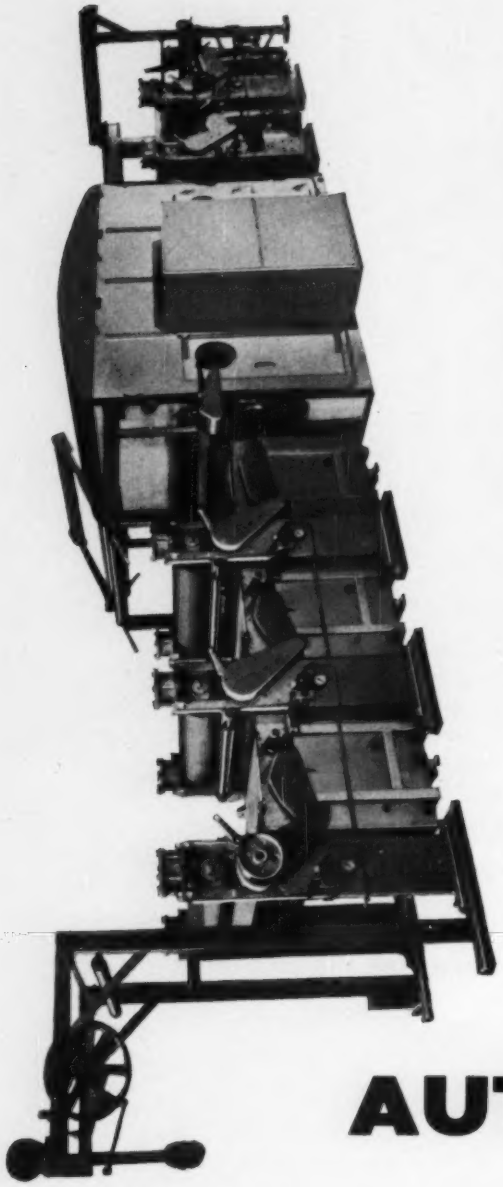

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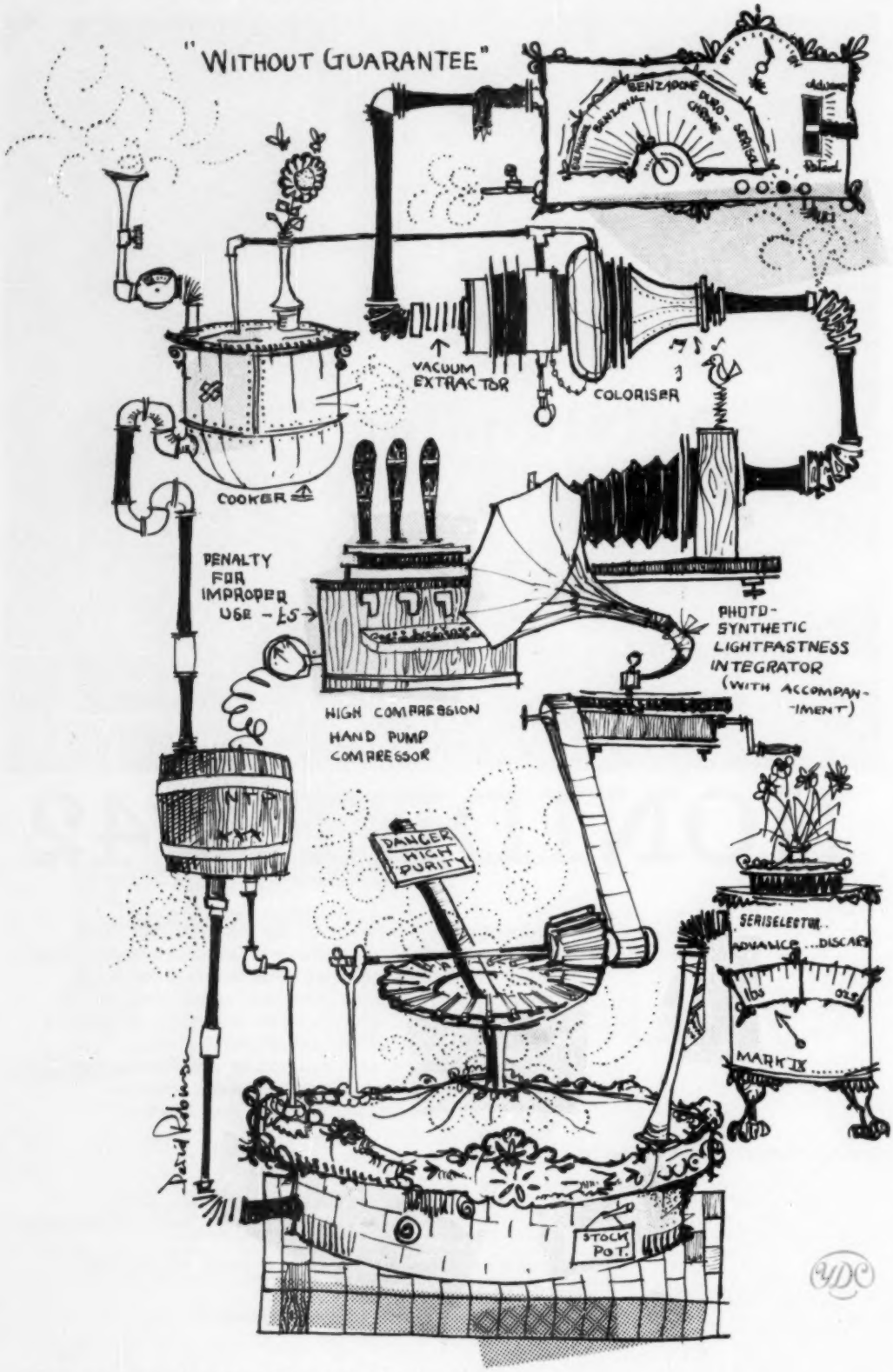

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Dr. Urquhart has provided a masterly chapter on sorption isotherms based mainly on data drawn from work on cellulosic fibres, although not everyone may wish to accept without some reservation his statement that general principles underlying the sorption process are, however, the same for all fibres.

Perhaps the most fascinating section of the book relates to the theories of adsorption, and it is most useful to have the theories of Peirce, Hailwood and Horrobin, and Brunauer, Emmett and Teller collected together with criticism and full references. There are other useful chapters by Meredith, on the effect of moisture on the mechanical and physical properties of fibres, by Hearle, on moisture and electrical properties, and by Crank, on diffusion. It is a feature of the book that definitions are given with great care and the whole work bears the stamp of authoritative treatment. It is a book that may be confidently recommended, both for reading and reference, to teachers and students and to all textile workers who value scientific principles.

G. LANDELLS

Lexikon Textilveredlung und Grenzgebiete

(Dictionary for Textile Finishing and Related Subjects)

By C.-H. Fischer-Bobsien. Pp. vi + 1193 + xv advert. 2nd Edition, Dülmen-Daldrup: Published by author. 1960. Price, DM 118.50 (to firms, technical schools, societies, etc. DM 93.50).

This dictionary of textile chemistry is already well established on the Continent, but the first edition, published some ten years ago, was not, unfortunately, brought to the notice of readers of the *Journal*, owing to the more difficult conditions obtaining at that time. It is important to rectify the omission, but at the same time to convey some idea of the scope of the work is no easy task for a reviewer. The author has interpreted the title in the broadest sense. About one thousand dye ranges are included, and the same number of natural and synthetic fibres. Application details for the former, and chemical and physical properties of the latter, are given in many cases. As an index of textile auxiliaries, some 3000 of which are mentioned, the book could stand alone.

Processes and machinery are not overlooked, and frequently line drawings are included for clearer exposition. As in the other items, the coverage is international, and well over a thousand organisations throughout the world have been consulted.

With a work of reference, such statistics are an impressive proof of the industry of an author, but cannot be taken as a certain indication of the usefulness of the book. In this case it must be said that most experienced textile chemists will find very little with which they are not already familiar, or at least they will already know where to find the information; but the convenience of having a considerable amount of data in a single volume was well brought out by presenting the book with random enquiries over several weeks. All were answered satisfactorily or good leads given.

As is to be expected, errors are rather frequent, but printed corrections are supplied. Also, some of the items listed are either trivial or are dealt with too cursorily. Again, the attempt to make the book a dictionary of foreign technical terms rather fails because in general the easy terms are chosen. On the credit side, the listing is alphabetical and hence a reader does not have to contend with the over-brief indexes of many German technical works; the language is simple and the book is well printed and bound. The price is rather high, but for anyone wishing to bring a small technical reference library up-to-date, this book could well serve the combined purposes of half-a-dozen other works and still show a measure of economy.

S. R. COCKETT

Appretur der Textilien: Mechanische und Chemische Technologie der Ausrüstung

By W. Bernard. Pp. viii + 440, with 274 figures in the text. Berlin, Göttingen, Heidelberg: Springer-Verlag. 1960. Price, DM 58.50.

The opening remarks by Herr Bernard in his introduction, if they apply to the literature of finishing in German, apply with greater force to the literature in English. He points out the lack of modern treatments of the subject, consequent on the developments in variety and detail that have taken place in the past few years. In this country we have, to a considerable extent, been supplied by the excellent work of J. T. Marsh. Even so, the very latest in plant and processes has scarcely achieved inclusion in text books, and the present work must be judged in the light of this situation. So viewed, it is a very able effort, comprehensive, up-to-date, well balanced, well arranged and well written.

An attractive feature is the wealth of illustrations. The line drawings of the working details of different machines are particularly clear and are well chosen to illustrate both the fundamental construction and modern improvement. The accompanying half-tones are also well reproduced. In the latter, the "new look" in structural features of modern plant is graphically brought out. To take two examples from many, the sections dealing with setting operations and with the various forms of impregnation pad-mangles are extremely interesting. Both stem from recent developments in man-made fibres or from the great extension of chemical finishes.

Further interesting sections deal with the chemistry of modern finishes and a wide selection of proprietary finishing agents, and finishing procedures are classified and explained.

On the whole, the sources most accessible to the author receive most attention. The greatest proportion of plant and products is German, but developments in other countries are not neglected, and few topics worthy of inclusion escape his attention. This is, therefore, a useful book and is to be recommended to all interested in finishing.

There is an adequate bibliography, together with selected lists of the manufacturers of plant and chemical products. As is usual with these publishers, the whole book is excellently produced.

H. A. TURNER

B.S. 3209 : 1960 Determination of Wool Fibre Medullation

Pp. 6. London: British Standards Institution. 1960. Price, 3s. 0d.

Many coarse wool fibres (average diameter > 25 μ) contain a tubular space known as a medulla. The effect of medullation is to lower the overall fibre density and to lighten the colour of the fibre because of reflection of light by the medulla.

This standard, based on T.T.S. No. 53 published in *J. Textile Inst.*, 1958, **49**, P 732, describes a method for determining medullation which is applicable to wool which can be arranged in bundles of a few hundred fibres suitable for making cross-sections. From measurements of cross-sectional areas of fibre and medulla, the percentage of medullated fibres and the volume percentage of medullation are determined.

M.T.

B.S. 3210 : 1960 Methods for the Analysis of Water-soluble Coal-Tar Dyes Permitted for Use in Foods

Pp. 34. London: British Standards Institution. 1960. Price, 7s. 6d.

B.S.I. is preparing specifications for the colouring matters permitted for use in foods under the Colouring Matter in Foods Regulations, 1957. As an essential step, methods of analysis applicable to water-soluble synthetic dyes are prescribed. Methods are given for the determination of matter volatile at 135°C, water-insoluble matter, di-isopropyl ether extract, subsidiary dyes, chloride, sulphate, dye content, copper, arsenic and lead. A limit test for heavy metals (as sulphides) is included.

M.T.

Style Guide for Chemists

By L. F. Fieser and Mary Fieser. Pp. vi + 116. New York: Reinhold Publishing Corpn., and London: Chapman & Hall Ltd. 1960. Price, 24s. 0d.

Very frequently nowadays one reads of the inability of scientists, among others, to communicate their ideas. Any book, therefore, that aims to remedy such deficiencies is to be welcomed. Style Guide for Chemists is, as its title implies, directed at one particular group of scientists, but much of its material is general in appeal.

The subjects discussed are, in order: concise writing (4 pp.), coherence (3 pp.), verbs (10 pp.), singular and plural forms (4 pp.), possessive singular (1 p.), emphasis (3 pp.), choice of words (14 pp.), punctuation (11 pp.), style (5 pp.), style sheet (12 pp.), proof (5 pp.), pronunciation (20 pp.), speaking (5 pp.). The index occupies 18 pp. It will be clear from this brief list that the approach is hardly logical; points of detail are discussed at once and such an important topic as the organisation of a paper does not receive mention until p. 51 is reached. In addition, for such a small book, the selection of material is unbalanced. According to both the Preface and the dust-jacket, the book is primarily one on writing, yet 20 pages are devoted to pronunciation, 15 of them to lists of words, names and prefixes. Each of these has a separate entry in the index, and entries such as "borneol, 80 (*pronunc.*)" account for much of its 18 pages.

Anyone writing a book for the guidance of others must exercise great care in both compilation and editing, for if the book is to be considered authoritative, practice must follow precept. The authors concede this, noting that "the words creep in, but they can be edited out. Learn what to look for, delete the offenders, and you will strengthen your writing and save space." Regrettably, this advice has not always been followed.

On p. 96 it is laid down that "colloquialisms are as out of place in a talk as in a paper". Yet at the foot of the same page we are told that "some little effort may be required . . . to *pep up* the story", and at the end of the chapter on Style (p. 55) we are exhorted to follow certain advice "not only at the beginning but *for keeps*". But perhaps these are colloquialisms only on this side of the Atlantic — which prompts the comment that the usage discussed is primarily American, so that British readers must use with care the lists of plural forms and the style sheet.

Other examples not attributable to possible trans-Atlantic differences in usage may be quoted. The first piece of positive advice on p. 1 is to "avoid needless words". The Preface, however, contains in two places the redundant phrase "a number of". Moreover, reference is also made here to "the latter two lists" (of lists of spelling, word-division, and pronunciation), but in the text proper (p. 20) it is stated that "former and latter are used when only two people or things are involved". On p. 21 an *alternative* is said to offer "*two* things, one of which must be chosen"; however, Fowler's *Modern English Usage*, which the authors have taken as their prime authority, gives as a definition "*set*, especially pair, of possibilities from which one only can be selected".

Further examples could be quoted to show that the book should be used with caution. This is unfortunate, for the material is attractively presented and well indexed.

M.T.

New Developments in Training

Edited by F. A. Heller. Pp. 80. London: Polytechnic Management Association. 1959. Price, 5s.

This is a group of five lectures first delivered in the Department of Management Studies in 1957 and since revised and edited.

The five contributors deal with: A Biologist's Contribution to Learning (Prof. J. Z. Young), New Methods of Training in Mechanical Skills (W. D. Seymour), Survey Findings in U.K. and European Countries (Mrs. W. Raphael), Training for Executive Skills (F. A. Heller), and Integration of Training, Organisation and Policy (S. D. M. King).

Taken together, these contributions appear to fulfil efficiently the general intention, viz., to be an introduction for an audience of managers and

industrial executives to general questions of work study and method organisation. Each contribution has features of considerable general and specific interest. The whole gives an insight for the layman into the methods that have developed in the past few decades for studying the complex problems of process analysis, operator training, and works organisation. Some of the lessons it describes could well be taken into account by those of us who are concerned with practical training in universities and similar institutions; occasionally, as is so common in this field, we may find with surprise that, like M. Jourdain, we have been speaking prose all our working lives. One emphatic statement by Mr. Heller—"the lecture method is inadequate and I am surprised that it has survived for so long even with the discussion period thrown in as an afterthought", is a challenge, and since it is followed by a review of other training methods, it could also stimulate general reform in technological teaching. Again, for the layman, the difficulties of setting out the data of studies of this kind so that precise conclusions can be drawn, are well illustrated. With analysis of skills and methods where numerical values can be assigned to performance, it is not difficult. Where more subtle human reactions and relations have to be discussed it is often much harder.

This book should be helpful to those candidates for our Associateship who intend to take paper E2. It might also help the Examiner!

H. A. TURNER

Technology of Non-metallic Coating

By A. Ya. Drinberg, E. S. Gurevich, and A. V. Tikhomirov. Oxford, London, New York, Paris: Pergamon Press. 1960. Price, 80s. 0d.

This is the English translation of a book originally published in Russia, late in 1957, for the use of chemical engineering students. It is concerned with the technology of the application of thin layers of non-metallic substances to metals and other substrates, e.g. wood, cloth, leather, and plaster, for protective and/or decorative purposes.

Chapter I deals with the corrosion of metals and its prevention; the electrochemical theories involved are given in some detail. Of the four preventive measures discussed, only one, the use of protective coatings, falls within the proper scope of the book. The next four chapters are: Properties of Painting Materials, and their Interaction with the Painted Surface; Scientific Principles of Coating Formation; Properties of Coatings and their Variety; Ageing of Coatings. These important matters are treated in terms of fundamental theory and technology, but sight is never lost of the relation to practical working.

The remaining 13 chapters mark this as, above all, a book for the practical man. They deal not only with the preparation and coating of various types of substrate in terms of both underlying theory and hard practice, but also with the equipment required. The profuse information includes many tables, graphs, diagrams, recipes, and flow sheets, and ranges from instructions for cleaning paint brushes containing squirrel, marten, or polecat hair, through thoroughly detailed calculations of the thermal characteristics of drying equipment (the subject of a separate chapter) and of the mechanical requirements of an overhead monorail conveyor (transport equipment has also a separate chapter), to the final chapter in which there is full discussion of the design and organisation of painting shops.

The chapter devoted to the coating of fibrous materials is of limited scope. After a rather superficial discussion of the interrelation between a woven fabric and an adherent coating, it deals at some length with lacquered fabrics for use in airframe construction (with a heavy emphasis on nitrocellulose), the impregnation of windings for electrical equipment, and the assisted surface finishing of leather—which occupies more than half of the chapter. There is no reference to vinyl-coated fabrics or to coated man-made fibres, both of which are of great importance and wide application. In another chapter, devoted to decorative and imitative finishes, textiles and paper are mentioned briefly, particularly in connection with the production of imitation suède (flock finishing), though no mention is made of the important method of electrostatic deposition.

The English is very readable and free from the unimaginative quaintness that bespeaks the pedantic or underinformed translator. Binding and paper are of good quality, and typographical errors are few—and unlikely to confuse anyone. There are many references to the literature and, though these relate in some instances to works published in Western countries, the majority are drawn from Russian sources and, consequently, are not readily accessible; this enhances the value of the book.

By virtue of its broad scope and its comprehensiveness in matters of technology and organisation, this book is of considerable interest and value to those engaged in surface coating in all its aspects. The price is not prohibitive, especially when one recalls how few books of any substance there are in this field.

J. W. DUARTE

Analytical Applications of Diaminoethanetetra-acetic Acid

By T. S. West and A. S. Sykes. Pp. 127. Poole: British Drug Houses Ltd. 2nd Edition. 1960. Price, 4s. 6d.

The first edition of this booklet was reviewed last year (J.S.D.C., **76** (Apr 1960) 240). The principal contents are unchanged, but an appendix (17 pp.) has been added and the list of references extended to a total of 523. Subjects discussed in the appendix are: primary standards, theory of end-point detection in complexometric titration, mathematical analysis of visual indicator end-point sharpness, visual indicators, developments in the use of conventional indicators, physico-chemical methods of end-point detection, masking action, the use of amalgams, miscellaneous applications of EDTA, newer complexones, and analysis of mixtures of complexones. In addition, a list of books recommended for further study is given.

Although the basic text remains unaltered, most of the formulae have been redrawn for greater clarity and where necessary [e.g. Di-SNADNS, p. 39 (2nd), 37 (1st)] corrected. As suggested by the original reviewer, *Colour Index* numbers have now been added, where appropriate, in the correct style. The reviewer also commented on the term Complexones; a footnote to the second edition states that I.U.P.A.C. (Mar 1960) has recommended that the term Complexan should be used instead.

This revision extends the usefulness of a very moderately priced book.

M.T.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index*

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W. T. Swatek

Melliand Textilber., **41** (Oct 1960) 1259–1263

Continuation of an article (*Ibid.*, **41** (June 1960) 746–749; J.S.D.C., **76** (Oct 1960) 596). The Pad–Roll and Pad–Steam systems are discussed in detail. The two systems can be combined for dyeing with direct and vat dyes. W.M.

PATENTS

Carbon Black (C.I. Pigment Black 7) Furnace

Phillips Petroleum Co. *BP* 857,256 (U.S.A. 8 May 1958)

Describes an improved form of furnace. C.O.C.

Wet Processing of Textiles in Rope Form

Maschinenfabrik Benninger *BP* 853,489 (Switzerland 3 Feb 1956)

The processing liquor is in a closed pressure container. There is at least one conduit for guiding the textile into and out of the container, there being a flow of a sealing fluid in the conduit during the operation of the machine. The guiding conduit forms or includes a throttle to increase the flow resistance of the sealing fluid and to build up the pressure in the container. This enables maintenance in the container of pressure greater than atmospheric. C.O.C.

Wet Treatment of Yarn and Cloth

J. Nüsslein *BP* 854,221 (Germany 7 Nov 1956)

The treatment is much more effective if the goods are closely wound on a perforated hollow cylinder having, preferably, both ends closed. The cylinder rotates rapidly and horizontally, the centrifugal force so generated forcing treating liquor supplied to the interior of the cylinder through the goods. C.O.C.

Paddle Machines for Wet Processing of Textiles

Freeman, Taylor Machines *BP* 854,619 (21 Jan 1958)

In a machine in which the goods are directly propelled around an annular space encircling an island to one side of which a multi-bladed rotary paddle is fixed, the inner portions of the paddle blades are articulated to members which pivotally connect them to blade-adjusting components. These blade adjusters can effect simultaneous radial projection or retraction of the blades. After adjustment they are locked together and to the connecting members to prevent relative turning movement between the components and the shaft. C.O.C.

Regulating the Tension of a Web during Processing

Benteler-Werke *BP* 853,157 (Germany 27 Nov 1957)

The cloth or other web passes alternately and successively over and under upper and lower rollers round which it is drawn by delivery rollers. The lower rollers are mounted on a cross member which can move up and down and which is connected to one arm of a double-armed pivoted lever. The other arm of the lever causes a counterbalance weight to provide compensation for part or all of the tensioning load imposed on the cloth by the lower rollers and their supporting cross-member. This renders possible uniform relief from drag of all the loops in the passage of the cloth with simultaneous appropriate regulation of the speed of the delivery rollers. C.O.C.

Screen Printing Machine

R. Kraft *BP* 854,794 (Austria 1 Oct 1957)

An endless stencil of material responsive to magnetic forces moves along the printing table. The material to be printed moves between the stencil and table at the same rate and in the same direction as the stencil. The table is constructed so that it can produce magnetic forces to attract the stencil to it. Magnetic rollers engage the outside of the stencil to guide it. This avoids all disadvantages of internal stencil rollers. The stencil may be of iron gauze, or iron powder may be added to the stencil varnish applied to silk gauze. C.O.C.

Vigoureux Printing Rollers

Ciba *BP* 855,588 (Switzerland 26 Apr 1956)

Having the impression elements detachably secured to the main body of the roller, e.g. by plates screwed on to the ends of the roller, cuts down the number of rollers that have to be kept available for use. C.O.C.

Curved Bar Cloth Stretching Machines

Calico Printers Engineering *BP* 852,367 (13 Dec 1957)

The cloth passes over at least three bars, some of which are driven and all of which are free to rotate. The position of the non-driven bars in relation to the driven bars depends on the warpwise tension in the cloth. Means are provided for causing the position of the non-driven bars to vary correspondingly and automatically with the speed of rotation of the driven bars and thereby maintain the warpwise tension of the cloth constant. C.O.C.

Automatic Screen Printing (IX p. 139)
Light-sensitive Coating for Preparing Printing Screens (IX p. 139)
Molten Metal Bath for Curing Plastics, Rubber, etc. (XIII p. 144)

II—WATER AND EFFLUENTS

Regeneration of Activated Carbon after Adsorption Purification of Effluents from the Synthetic Dye Industry

L. A. Kal'skiĭ, A. M. Koganovskiĭ, E. M. Kaliniĭchuk, and E. I. Dikolenko *Khim. Prom.*, (4) (1959) 46–49

Chem. Abs., **54** (10 Nov 1960) 23121

A detailed account of the steam-regeneration of various types of activated carbon. Differences in the composition of the effluents has little effect on the quality of the regenerated carbons. C.O.C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Effect of Surfactants and Protective Colloids on the Stability of Hydrophobic Sols at High Electrolyte Concentration

H. Lange *Kolloid-Z.*, **169** (1960) 124–131

Chem. Abs., **54** (10 Nov 1960) 21935

The effect of surfactants on the stability of paraffin sols depends to some extent on alteration of the electric surface potential, but largely on formation of hydrated layers on the surface of the sol particles. The action of these layers is clearly shown by the ability of the surfactant to prevent coagulation of sols by electrolytes, even when the concentration of electrolyte (NaCl) is so high as to eliminate the electrostatic repulsion between the particles.

C.O.C.

Reagents for Crease-resist Processing

J. T. Marsh *Text. Rec.*, **78** (Dec 1960) 52–54

The composition of various types of bi-functional reagents and their probable reaction mechanism with cellulose is discussed, including wet cross-linking, as in the Belfast finish of Deering Milliken. W.G.C.

PATENTS

Mordants for Acidic Organic Compounds

Gevaert Photo Producten *BP* 852,483 (Holland 8 Mar 1957)

The products obtained by treating polymers containing nitrile groups with hydroxylamine at 40–120°C have strong mordant properties for acidic compounds. Thus they may be used to prevent the diffusion of acid dyes in photographic emulsions. C.O.C.

Lubricant for Polyester Fibres

N.V. Onderzoekinginstituut Research *BP* 853,179 (Holland 15 Jan 1957)

Polyester fibres and yarns are lubricated for processing by treating them with a 0·5–10·0% aqueous solution of an alkali-metal salt of an acid of dissociation constant $> 10^{-9}$ at 20°C, e.g. Na_2CO_3, Na_3PO_4, and Na stearate, rinsing and drying. It is preferable that the treatment be at > 60°C. C.O.C.

Detergents

Henkel & Cie. *BP* 858,239 (Germany 10 Oct 1956)

Excellent detergents unaffected by hard water are obtained by treating salts of surface-active *N*-acylaminocarboxylic acids with 1–20 mol. of ethylene oxide. C.O.C.

Scouring and Milling Agent

Deering Milliken Research Corpn. *USP* 2,925,639 (30 Nov 1953)

A scouring and milling agent which can be used under neutral and acid conditions consists of an aqueous mixture of a detergent which is more hydrophilic than lipophilic, e.g. Tergitol NPX (CCC), and a wool plasticiser, preferably cationic, e.g. Ahcovel G (AH). C.O.C.

Phenylphenol Compositions as Assistants in Dyeing Hydrophobic Fibres with Acid and Disperse Dyes

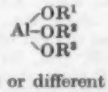
General Aniline *USP* 2,926,987 (30 Apr 1956)

o- or *p*-Phenylphenol is mixed with a dispersing agent, e.g. Tamol NNO, in presence of a little water, the resulting powder heated until all the water is removed, and then finely ground in a hammer mill. The product is an excellent assistant in the application of disperse dyes to polyester fibres or acid dyes to acrylic fibres. C.O.C.

Water-repellent Composition for Application from an Organic Solvent

Gy *BP* 853,765 (26 May 1956)

A solution in a hydrocarbon and/or chlorinated hydrocarbon of a mixture of a hydrocarbon wax of m.p. 30–90°C and a compound of formula—



(R^1, R^2 and R^3 = same or different aliphatic of 3–30 C), e.g. Al isopropylate, gives a greater water-repellent effect than a mixture of wax and solvent, or of wax, solvent, and Al soap. C.O.C.

Quaternary Ammonium Thioglycollates

Glovers (Chemicals) *BP* 859,770 (29 May 1956)

Compounds of formula—

$$R^1R^2R^3R^4N^+O^-OCCH_2SH$$

(R^1, R^2, R^3 and R^4 = same or different, Alk or hydroxyalkyl and together are of > 9 C), e.g. hexadecyltrimethyl ammonium thioglycollate. They are useful as bactericides, textile finishing agents and in the permanent setting of keratinous fibres. C.O.C.

Rotproofing Agents

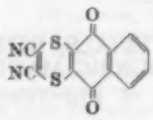
ICI *BP* 852,634 (23 Jan 1956)

αα′-Dichloromaleimides substituted on the N atom by $-(CH_2)_nR$ (R = monocyclic carboxylic group; n = 0 or 1) are effective fungicides and rotproofing agents. Thus *N*-(4-methylphenyl)dichloromaleimide is an effective rotproofing agent for cotton and has good resistance to leaching. C.O.C.

Fungicides

E. Merck *BP* 857,383 (Germany 26 Oct 1957)

2,3-Dicyano-1,4-dithia-anthrahydroquinone and the corresponding quinone—



are stronger fungicides than those hitherto on the market. C.O.C.

Thiourea Resins for Rendering Nylon Flame-resistant

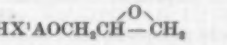
American Cyanamid Co. *USP* 2,922,726 (18 Mar 1955)

0·4 Mol. of a water-soluble aliphatic monohydric alcohol and 1 mol. (on a monomeric basis) of a hydrophilic thiourea–aldehyde condensate are heated together at > 45°C at pH 4–6. The reaction is stopped when much of the alcohol has reacted and before a sample of the reaction mixture hydrophobes when diluted with 50 volumes of water at 20°C. A mixture of the product and a hydrophilic water-soluble monomeric or low polymer of methylolurea or a lower alkyl ether thereof, the proportion of the mixture being such that there is 0·6–7·0 parts by wt. of urea to 1 of thiourea, is used with a halide catalyst to produce a durable fire-retardant stiff finish on nylon net. C.O.C.

Unsaturated Glycidyl Ethers and their Polymers

Rohm & Haas Co. *BP* 853,619 (U.S.A. 6 Feb 1957)

Describes production of compounds of formula—



[X^1 = O or S; A = alkylene of 2–12 C having at least 2 C between the O and X atoms and which may or may not be substituted by cycloalkyl, Ar or aralkyl, or alternatively A = $-(C_nH_{2n}X^2)_xC_nH_{2n}-$ (X^2 = O or S; n = 2–12 and x = 1–5)]. Their polymers have a very wide range of uses, including the dimensional stabilisation of wool, cotton, and rayon cloths, crease-shedding finishes, moulding powders, etc. Thus a copolymer of n-butyl acrylate (95 parts by wt.) and 2-(vinylthio)ethylglycidyl ether is used in aqueous dispersion to impart a shrink-resisting finish to all-wool flannel. C.O.C.

Stable Antivesicants for Impregnating Cloth

U.S. Secretary of War *USP* 2,926,107 (29 Mar 1933)

2,4,6-Trichlorophenylbenzoyl chloramine and 2,4,6-trichlorophenylacetylchloramine are very stable antivesicants when impregnated into cloth. C.O.C.

Chlorinated Phenol Composition for Impregnating Cellulosic Materials

Monsanto *USP* 2,926,096 (27 Apr 1955)

Concentrated solutions of chlorinated phenols are obtained by using a phosphoric ester as the sole or larger part of the solvent. These solutions are readily diluted with volatile solvents. There is no blooming of the treated material. C.O.C.

Solutions of Acrylonitrile Polymers

Chemstrand Corpn. *BP* 856,315 (U.S.A. 30 Dec 1957)

A mixture of chloral hydrate and succinonitrile, malononitrile or a cyclic alkylene carbonate (alkylene of 2–4 C), e.g. ethylene carbonate, dissolves acrylonitrile polymers. The solutions are stable at room temperature to 45°C. C.O.C.

Gluconic Acid as an Antirusting Agent

ICI *BP* 855,442 (22 May 1957)

Gluconic acid and functional derivatives thereof, e.g. D-glucono-δ-lactone, are antirusting agents for use in aqueous preparations. C.O.C.

Affinity for Cellulose of Polyenedicarboxylic Acids of the Crocetin Type and of Quaternary Ammonium Compounds (VI p. 133)

Rates of Adsorption of Sulphuric Acid on Wool in Presence of Surface-active Agents (VI p. 134)

Preparation and Use of Peracetic Acid in Bleaching (VII p. 136)

Stabilisers for Alkaline Peroxide Bleaching Baths (VII p. 136)

Antioxidants giving Protection against Ozone Cracking (XIII p. 144)
Flameproof Composition for Application to Plastics and Resins (XIII p. 144)

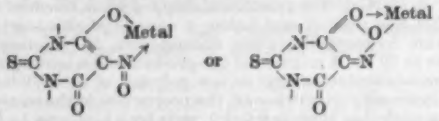
IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Metal Complexes of Di-*p*-Tolylthiovioluric Acid

R. P. Singh

J. Indian Chem. Soc., 37 (Sept 1960) 569–572

Highly coloured metal chelates of the title acid are now reported. In a table are given the colours, decomp. temp., λ_{max}, % sulphur, % metal, and metal : acid ratio of 38 metal complexes. It seems highly probable that the rings in the complexes are six-membered, but no decision has been arrived at with respect to the alternative structures possible, viz.—



since the co-ordinating power of N or of negative oxygen O^- is roughly the same (cf. Sidgwick, *J.C.S.*, (1941) 433).

H.H.H.

Studies on Coupling Reaction of α-Naphthol—I

Y. Bansho and I. Saito

Rept. Govt. Chem. Ind. Research Inst. Tokyo, 55 (Sept 1960) 337–345

Reaction of α-naphthol and aniline yields 4-phenylazonaphthol (I), 2-phenylazonaphthol (II), and 2,4-bisphenylazonaphthol (III). At pH < 10, the main product is I; above this pH III predominates; the yield of II is almost independent of pH. I couples further with the diazonium salt to give III, but II does not. M.T.

^{14}C-labelled Dyes by way of Benzene from Acetylene-^{14}C

H. Baddenhausen, H. Götte, and L. Wiesner

Angew. Chem., 71 (1959) 444–445

Chem. Abs., 54 (10 Nov 1960) 23335

Describes a micro method developed for the cyclisation of acetylene-^{14}C to benzene-^{14}C according to Reppe and Schweckendieck's method (*Chem. Abs.*, 43 (1949) 6202). Benzene-^{14}C was nitrated, reduced with Sn dust, and the resultant amines separated as the hydrochlorides. The amines were then separated by paper chromatography according to Ekman (*Chem. Abs.*, 43 (1949) 2263). Simple dyes, e.g. 2,4-diaminoazobenzene-HCl or *p*-dimethylaminoazobenzene, were prepared *in situ* on the chromatographic paper, and the resultant dyespots cut out and eluted. The yields of labelled dyes were only 15%, related to aniline, because diazotisation was not carried out at the necessary low temperature. C.O.C.

Azo Dyes from Acetanilidovinyl Heterocycles

H. Wahl *Compt. rend.*, 250 (1960) 2908–2909

Chem. Abs., 54 (10 Nov 1960) 22576

To 1-methyl-2-acetanilidovinylbenzothiazolium methosulphate (I) (4·20 g) in aq. pyridine was added an equimolecular amount of 4-chlorobenzenediazonium chloride in water at 5–10°C. This precipitated the orange-yellow dye 8-(4′-chlorophenylazo)-8-formyl-2-methylene-1-methyldihydrobenzothiazole. I with 4-nitrobenzene diazonium chloride gave 8-(4′-nitrophenylazo)-8-formyl-2-methylene-1-methyldihydrobenzothiazole. With the corresponding benzoselenazole there was obtained 8-(4′-nitrophenylazo)-8-formyl-2-methylene-1-methylbenzoselenazole. C.O.C.

Azo Dyes from Sulpha Drugs

P. P. T. Sah, J. F. Oneto, and H. Sah

Arzneimittel-Forsch., 10 (1960) 533–540 (*in English*)

Chem. Abs., 54 (10 Nov 1960) 23195

Preparation of 36 dyes by diazotising any of 6 sulpha drugs and coupling with itself and each of the other 5 is described, e.g. sulphanilamide was diazotised and coupled with itself or sulphapyridine. The azo group always coupled in the 3-position of the sulphanilamide ring. C.O.C.

Oxidation of Aminoaryl Derivatives of Naphtho-[1,2]Triazole

J. Haase

Collection Czech. Chem. Communs., 25 (1960) 716–728

Chem. Abs., 54 (25 July 1960) 14199

Subst. naphtho[1,2]triazoles were oxidised with Na hypochlorite and the complex mixtures obtained separated by chromotography (Al_2O_3). Thus, dyes were obtained from 2-*p*-aminophenyl-6,8-disulpho-, 2-*p*-aminobiphenylyl-6,8- and -4,7-disulpho-naphtho[1,2]triazoles, e.g., giving 4,4′-bis[2-(6,8-disulphonaphtho[1,2]triazolyl)]-azobenzene. Oxidation of 2-(*p*-aminophenyl)naphtho[1,2]triazole gave a mixture of 4,4′-bis-(2-naphtho[1,2]triazolyl-azo(I), 2-chloroazo(II), 2,6-dichloroazo(III)-benzenes and 2,7-bis(2-naphtho[1,2]triazolyl)phenazine (IV). II and III reduced with $Ti_2(SO_4)_3$ gave a mixture of I, 2-(3-chloro(V)- and 3,5-dichloro (VI)-4-aminophenyl) naphtho[1,2]triazole. Diazotisation of 1-acetamido-2,6-dichloro-4-aminobenzene and coupling to β-naphthylamine gave acetyl-VI and similarly acetyl-V was prepared. A series of derived dyes of this type is fully described, various methods of synthesis being used. A.T.P.

Absorption Spectra of Dyes. IX—Changes of Absorption Curves caused by Exposure of Aqueous Solutions of Dyes to Light

Y. Ishii and M. Shimada

J. Soc. Textile Cellulose Ind. Japan, 16 (July 1960) 579–583

Aqueous solutions of Benzo Azurine G (C.I. 24140), its Cu complex, and Congo Red (C.I. 22120) were exposed to a mercury quartz lamp and to a carbon arc lamp. To utilise both the visible and ultraviolet radiation from the quartz lamp two filters were used. The results showed that the Cu-complex dye of high fastness to light in regenerated cellulose film showed no appreciable difference in decrease of optical density in aqueous solution compared with C.I. 24140. The selected light (λ = 3000–4000 Å), faded aqueous solutions of the two dyes to the same degree, the other light faded C.I. 24140 more readily than its Cu-complex. Congo Red fades more rapidly under the carbon arc than do C.I. 24140 and its Cu-complex. C.O.C.

Preparation of 1,5- and 2,6-Chloronaphthols

N. N. Vorozhtsov and V. N. Lisitsyn

Zhur. obshch. khim., 30 (Sept 1960) 2816–2817

On heating 1,5-(or 2,6-)dichloronaphthalene with caustic soda in methyl alcohol in presence of copper, the corresponding naphthols are obtained. During synthesis of 1,5-dichloronaphthol, a certain quantity of the methyl ether is also obtained. L.S.L.

Fastness to Light of Triphenylmethane Basic Dyes

V. F. Borodkin *Trudy Ivanovsk. Khim. Tekhnol. Inst.*, (8) (1958) 116–117

Referat. Zhur. Khim., (1959) Abstr. 39622

Chem. Abs., 54 (10 Nov 1960) 23335

Study of the fastness to light of triphenylmethane basic dyes containing Cl, F, OH, NO_2 or sulphoxido groups *ortho* to the central C-atom showed that an increase in fastness was imparted by these substituents. Depending on the nature of the substituent, fading was slow or rapid, but always quicker than with the unsubstituted dyes. C.O.C.

Association in Concentrated Solutions of Dyes as shown by their Absorption and Luminescence Spectra

V. L. Levshin, E. G. Baranova, L. D. Derkachëva, and L. V. Levshin

Termodinam. i Stroenie Rastvorov, Akad. Nauk S.S.S.R., Otdel. Khim. Nauk, Khim. Fak. Moskov. Gosudarst. Univ., Trudy Soveschaniya, (1958) 275–285

Chem. Abs., 54 (10 Nov 1960) 21942

Study of the spectra showed that the degree of association of Rhodamine 6G and 3G (C.I. Basic Red 1 and 3) increased with concentration. Spectra of aqueous soln. of 3,3′-diethylcarbothiacyanine chloride indicate rapid formation of higher associations. At all degrees of association studied, absorption at λ = 575 mμ remained constant. The ultraviolet spectra of aqueous solutions of rivanol (7-ethoxy-2,5-diaminoacridine) yielded curves passing through the same point. Spectra of aqueous

solutions of Safranine (C.I. Basic Red 2) indicate that higher concentration results in increased association. The spectra of aqueous solutions of pseudocyanine indicate that the molecules forming higher associations interact only weakly with one another. Mixed associations of 6G with 3G, and of 6G with Crystal Violet (C.I. Basic Violet 3) and other dyes are less stable than homogeneous associations. Mixed associations of different structure and composition can be present. The quenching of fluorescence in 5% aq. NaCl soln. is predominantly caused by inactive absorption and in alcoholic solution because of transfer of the energy to associated molecules. In aqueous solution both mechanisms are commensurable. Solutions of pseudocyanine became luminescent when larger associations are formed, the ν of luminescence depending on the size of the aggregates. Increase in temperature leads to disaggregation. In studying this effect a correction for the "temperature" quenching has to be made. Associations of pseudoisocyanine are unstable. The intensity of luminescence of its solutions was reduced 40-fold by a rise in temperature of 20 degrees C. C.O.C.

Optical Properties of Concentrated Solutions and Melts. I— Solutions of Dyes

V. A. Kizel and V. M. Rubinov

Optika i Spektroskopiya, **7** (1) (1959) 62–70
Chem. Abs., **54** (10 Nov 1960) 21997

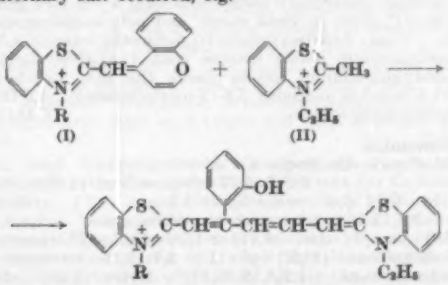
It is possible to use the method of selective reflection to determine the optical constants in the region of the absorption band of conc. soln. of dyes. Absorption, reflection, and dispersion curves of Methylene Blue (C.I. 52015), nitrosodimethylaniline, Crystal Violet (C.I. 42555), Rhodamine 6G (C.I. 45160) and Fuchsine (C.I. 42510) in conc. soln. and bromine are best described by the Davidov theory (*Chem. Abs.*, **50** (1956) 11085) for the case of strong interaction of the vibrations of the molecules of both solute and solvent (aniline). Use of the above theory enabled the force constants of the oscillators and frequencies of electronic transitions to be determined. The results agreed with those obtained by other methods and in other solvents. C.O.C.

Synthesis of Dicarbocyanines by Splitting Pyran Ring in Pyrancyanines

A. I. Tolmachev

Zhur. obshch. khim., **30** (Sept 1960) 2892–2904

When monomethin cyanines containing a 4-benzopyran nucleus (I) reacted with nitrogen-containing quaternary salts containing an active methyl group (II), splitting of the pyran ring with simultaneous attachment of the quaternary salt occurred, e.g.—



The resulting dicarbocyanines contain *o*-hydroxyphenyl substituents in the chromophores. A similar reaction will occur with monomethin cyanines containing 4-benzothiopyran and 4-α-naphthopyran residues. Absorption spectra of the synthesised dyes were measured. L.S.L.

Thiobarbituric Acid Methin Dyes

H. Schmidt

Fette, Seifen, Anstrichmittel, **61** (1959) 881–886

The designation TBS-dye 532 is preferred for the red dye described in *Chem. Abs.*, **53** (1959) 22957 and having ϵ_{max} at 532 mμ. Originally obtained from oxidised fats, it can also be prepared from substances which, on oxidation, acid hydrolysis, or treatment with alkali, yield malonic aldehyde or β-hydroxyacrolein, which react with thiobarbituric acid. A structural formula is given for this dye and also for other trimethin dyes prepared and having ϵ_{max} at 457, 622, 549, and 542 mμ. 21 References. C.O.C.

Pentamethin Pyridine Dyes

N. E. Grigor'eva

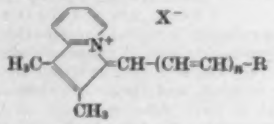
Uchenye Zapiski, Khar'kov. Gosudarst. Univ. A. M. Gor'kogo, Trudy Khim. Fak. i Nauch.-Issledovatel Inst. Khim., **95** (18) (1957) 207–230
Chem. Abs., **54** (10 Oct 1960) 19667

Study of the relation between constitution and colour, and the condition and nature of the conversion of pyridine dyes by heat. Over 40 known symmetrical dyes were made by the Zincke method, and their constitutions and those of the products of their decomposition on heating were studied. Decomposition by heat took place according to $[ArHN(CH:CH)_3CH:NHAr)]^+Cl^- \rightarrow (C_5H_5NAr)^+Cl^-$ (I) + $ArNH_2$. Addition of Ar to N in the pyridine ring shifted the electron distribution and made the C–C linkages more unsaturated, so that I is hydrogenated more easily than C_5H_5N. Degradation of dyes of formula—

$(XC_6H_4C_6H_4NH(CH)_5{:}NHC_6H_4C_6H_4X)^+Cl^-$

(X = NH_2, H, NO_2) (II) confirmed this. II cyclised on heating without decomposition and were stable up to 280–300°C, and to alkalis and acid. Action of alkali on pyridine dinitrochloroarylates (III) gave red glutaconaldehyde monoanils of possible structure ArNHCH:CHCH:CHCHO (IV) or ArN:CHCH:CHCH:CHOH. Alkali with *p*-subst. pyridine chloroarylates, $(C_5H_5NC_6H_4X)^+Cl^-$ (X = H, CH_3, OH, NH_2, COOH, SO_3H, NO_2) formed yellow compounds turning red on heating because of opening of the pyridine ring. Cleavage of the ring depended on the *p*-substituent, the chance for ring cleavage diminishing with increase in the electron-donating character of the group. Primary amines reacted with 2,4-dinitrochlorophenylate and added to the C atom of least saturation in the pyridine ring. Strong bases formed dinitroaniline and the symmetrical dye RN:CHCH:CHCH:CHNHR. The picolines formed with 2,4-dinitrochlorobenzene salts similar to those from pyridine which were split by alkali and amines to the pentamethin dyes having the CH_3 in the side chain. The dinitromonoanil reacted in acid with aromatic primary and secondary amine to form pyridine dyes. Other, similar monoanils of IV do not react with aromatic amines because of H-bond formation between N and O. However, they reacted with other compounds. IV and 2-methylbenzothiazole in acetic anhydride formed hexamethin dyes. The colour change of IV (Ar = C_6H_5, $pCH_3C_6H_4$, $pCH_3OC_6H_4$, $C_6H_5C_6H_4$, *p*-HOC_6H_4, 2,4-$(NO_2)_2C_6H_3$, $C_6H_5CH_2N$, α- and β-naphthyl) in methanol, ethanol, butanol, acetone, pyridine, dichloroethane, acetic acid, dioxan, benzene, bromobenzene, and nitrobenzene and in presence of alkalis and acids was observed earlier but the cause of change was not clear. The visible and ultraviolet absorption spectra of these solutions showed no direct relation between colour change and polarity of the solvent. The colour change was connected with formation of molecular compounds between IV and solvent. The colour change in presence of acids depended on the dielectric constant of the solvent, colour intensity increasing as the dielectric constant decreased. Intensity of colour in alkaline medium decreased with increase in basicity of IV. Spatial arrangement of the atoms in the molecule and opening up of the pyridine ring in the reaction of pyridine arylates with primary or secondary amines had profound influence on the intensity of the colour. Primary amine derivatives, and condensates with the pyridine ring opened, were more strongly coloured than the secondary amine derivatives and condensates retaining the cyclic pyridine ring. Shift of maximum absorption from 485 mμ for the anil derivative to 450 mμ for the *N*-methylanil is caused by destruction of the coplanar arrangement of the benzene ring in the latter. Substitution of an electron-donating group in $YC_6H_4{:}NC_5H_4NH_2C_6H_4X$ (X = H < CH_3 < OCH_3 < OH < NH_2 ≤ N $(CH_3)_2$) and an electrophilic group (Y = SO_3H < SO_2NH_2 < COOH < $COOC_2H_5$ < NO_2) increased the intensity of colour. Introduction of NO_2 groups in the anion had a similar bathochromic effect to $N(CH_3)_2$ in the cation of similar structures. The wavelength of maximum absorption of 4,4′-dinitropyridine dyes, 527 mμ, was nearly the same as that of the corresponding amino-substituted derivative (530 mμ), but the intensity of colour of the alcoholic solution was nearly 4 times greater with the latter. A nitro group strongly affected the intensity of colour, but had little effect on the shift in absorption, whereas $N(CH_3)_2$ influenced both. C.O.C.

Polymethin Dyes of the Indolazine Series
F. N. Stepanov and L. I. Lukashina
Zhur. obshch. khim., **30** (Sept 1960) 2850–2853
Tri- and penta-methin dyes of formula—



where R = indolazine or aniline, n = 1 or 2, X = Br or ClO_4, have been prepared and their absorption spectra in the visible region measured. L.S.L.

Synthesis of Neocyanine Dyes of the Brooker-type and Related Dyes
S. Kimura

I— Examination of Previous Process for Synthesis of Brooker-type Dyes
Bull. Soc. Sci. Phot. Japan, (9) (Dec 1959) 29–40
The dye assumed by Tanabe (*Chem. Abs.*, **48** (1954) 8093) to be 1,1′,1″-triethyl-10[2-(2″-quinolyl)vinyl]-quinocarbocyanine-1-chloride-1″-iodide is shown to be a double compound of Pinacyanol and structurally unidentified white crystals, m.p. 158–161°C. The dye assumed by Tanabe to be 3,3′,3″-triethyl-9[2-(2″-benzothiazole)-vinyl]thiacarbocyanine di-iodide (I) is actually a double compound of 3,3′-diethylthiacarbocyanine iodide and a structurally unidentified white crystalline compound. Apparently Tanabe's so-called Brooker-type dyes are a complex of an intermediate compound and a carbocyanine formed from a vinylaniline derivative of the heterocyclic quaternary ammonium salt used as the other intermediate.

II— Synthesis of the Brooker-type Dyes
Ibid., 41–44
Chem. Abs., **54** (10 Nov 1960) 22113
Describes the synthesis of the green 3,3′-diethyl-3″-methyl-9[2-(2″-benzoselenazolyl)vinyl]-thiacarbocyanine di-iodide and an unidentified purple dye. C.O.C.

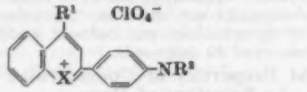
Indigoid Vat Dyes of the Isatin Series. X— (3-Indolyl)(5-methoxy-2-thianaphthenyl) Indigo-type Dyes
S. K. Guha, J. N. Chatterjea, and A. K. Mitra
Chem. Ber., **93** (1960) 1925–1927
Chem. Abs., **54** (10 Nov 1960) 23335
Title dyes from 3-hydroxy-5-methoxythianaphthene (I) and various isatins are more deeply coloured than the corresponding 5-CH_3 and 5-Hal derivatives. Preparation of several such dyes is given. C.O.C.

Preparation of Copper Phthalocyanine (C.I. 74160) from Phthalic Anhydride
V. F. Borodkin
Trudy Ivanovsk. Khim. Tekhnol. Inst., (8) (1958) 101–104
Referat. Zhur., Khim., (1959) Abstr. 39627
Chem. Abs., **54** (10 Nov 1960) 22676
Study of the formation of Cu phthalocyanine from phthalic anhydride, urea, and Cu_2Cl_2. A cupriferous di-iminoisoindoline was isolated and its properties determined. A cupriferous di-iminoisoindoline was made from di-iminoisoindoline and Cu_2Cl_2. Mechanism for the intermediary formation of di-iminoisoindoline during the formation of Cu phthalocyanine is suggested. C.O.C.

Synthesis of Solvent Dyes. IV— Sulphonated Copper Phthalocyanine Dyes
S. Hashimoto and Y. Isaka
Dōshisha Kōgaku Kaishi, **8** (1957) 130–134
Chem. Abs., **54** (10 Nov 1960) 23335
Cu phthalocyanine dyes were prepared according to *BP* 464,124, and sulphonated by Carleton and Woodward's method (*Chem. Abs.*, **33** (1939) 5671). C.O.C.

Action of Complex-forming Additives in Synthesis of Phthalocyanines. I— Influence of Phosphoric Acid Derivatives and Molybdenum Trioxide on the Synthesis of Fe Phthalocyanine
A. P. Rudenko and H. P. Dobrosel'skaya
Zhur. obshch. khim., **30** (Sept 1960) 3077–3083
Additions of small quantities of molybdenum and phosphorus in the form of orthophosphoric acid, ammonium dihydrogen phosphate, molybdenum trioxide or ammonium molybdate decrease the rate of formation of Fe phthalocyanine, whilst similar additions of heteropolycompounds, e.g. phosphomolybdic acid and ammonium phosphomolybdate, increase the rate of reaction. The catalytic action of the additives occurs during phthalamide condensation (formation of C-N linkages). The mechanism of formation of Fe phthalocyanine from phthalamide and iron via multi-stage polycondensation of phthalamide is suggested. L.S.L.

Condensation of Tertiary Oxonium Salts of Chromones and Thiochromones with Dialkylanilines
A. I. Tolmachev
Zhur. obshch. khim., **30** (Sept 1960) 2884–2892
Tertiary oxonium salts of chromones and thiochromones will react in acetic anhydride with dialkylanilines to form 4-methoxy- or 4-methylmercapto-4′-dialkylaminoflavones of general formula—



where X is O or S, R^1 alkoxy, R^2 ethyl or methyl. These in turn condense readily with compounds containing active methyl or methylene groups to form dyes. L.S.L.

Fluorescent Derivatives of 1,2,3-Triazoles
J. Dobáš and J. Pirkl
Collection Czechoslav. Chem. Communs., **25** (1960) 912–918
Chem. Abs., **54** (10 Nov 1960) 22602
Study of the colour and fluorescence of a series of 2-phenylnaphtho[1,2]triazole-6-sulphonic acid derivatives. Electrophilic groups in the 4′-position behaved as auxoflores, whereas the electron donor groups OH and NH_2 in the same position behaved simultaneously as auxochromes, bathoflores, and diminoflores. Electron bond groups in 3′-position quenched the fluorescence in aqueous solutions, the electrophilic groups caused no change in fluorescence, and both series of substituents had no effect on the colour. The causes of these phenomena are discussed. C.O.C.

PATENTS

Oxazolo[4,5-f]benzothiazoles as Dye Intermediates
General Aniline *BP* 853,724 (U.S.A. 25 Feb 1957)
Oxazolo[4,5-f]benzothiazoles containing Alk or aralkyl in the 2-position and Alk, Ar or aralkyl in the 6-position are readily quaternised and then treated with cyclo-ammonium quaternary salts containing a reactive group in the α-C atom to yield mono- and poly-methinecyanine dyes. Thus 3-carboxymethyl-2,6-dimethyloxazolo[4,5-f] benzothiazolium iodide treated with 1-ethylmercapto-6-methoxyquinolinium iodide yields the dye 3-carboxy-ethyl-3′-ethyl-6′-methoxy-5,6-[2-methyloxazolo 4,5]thia-pseudocyanine iodide. C.O.C.

Carotenoids
F. Hoffmann–La Roche & Co.
BP 859,731 (Switzerland 14 Feb 1958)
1,18 - Di[2′,6,6′ - trimethylcyclohexen - (2′) - ylidene - (1′) - 3,7,12,16 - tetramethyloctadecahexaen - (2,4,6,12, 14,16) - yn - (9) - diol - (8,11) or 1,18 - di[2′,6′,6′-trimethyl-cyclohexadiene - (1′,3′) - yl - (1′) - 3,7,12,16 - tetramethyl-octadecahexaen - (1,3,5,13,15,17) - diyn - (8,10) - diol-(7,12) is treated with a lower alkanoic acid, the resultant product hydrolysed and then oxidised with Al alkoxide and a ketone. After either of these steps one or two triple bonds are partly hydrogenated to double bonds in presence of a hydrogenation catalyst which permits of interruption of the hydrogenation at the olefinic stage or which permits hydrogenation only to that stage. Any di-*cis* compounds obtained by this hydrogenation may, if desired, be isomerised to the all-*trans* compounds. C.O.C.

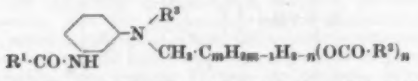
Yellow Solvent Dyes for Colouring Petrol
Eastman Kodak Co. *USP* 2,925,333 (24 May 1955)
The yellow dyes obtained by coupling diazotised *p*-nitroaniline with *N*-s-butyl aniline, *N*-isobutyl aniline, *N*-butyl aniline, or *N*-ethyl-*o*-toluidine have high tinctorial value in petrol and other hydrocarbons, more than three times as great as that of *NN*-dimethyl-*p*-phenylazoaniline. They have the further advantage of being soluble in tetra-ethyl lead mixes. C.O.C.

Monoazo Disperse Dyes for Nylon and Polyacrylonitrile Fibres
ICI *BP* 856,899 (4 Oct 1957)

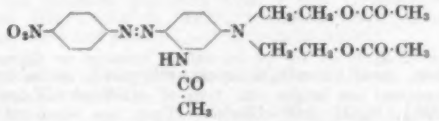
Disperse dyes containing alkoxy, or preferably alkylamino, groups substituted on a C atom or atoms by Cl or Br, or by OH and Cl or Br, or by an epoxy group have good wet fastness on nylon and modified acrylonitrile fibres, probably due in part to chemical reaction between dye and fibre. Thus application of the monoazo compound *p*-nitroaniline → *NN*-bis(γ-chloro-β-hydroxypropyl)aniline to nylon at 95°C, followed by treatment in hot aq. Na_2CO_3, gives a red of good fastness to washing. E.S.

Monoazo Disperse Dyes
S *BP* 856,348 (Switzerland 25 July 1956)

Disperse dyes similar to those of *BP* 819,644 (J.S.D.C., **75** (1959) 618) are prepared by coupling suitable diazo compounds with *O*-acylated *N*-hydroxyalkyl derivatives of *m*-aminoacylanilides—



(R^1, R^2 = CH_3 or C_2H_5; R^3 = Alk of 1–4 C or $-CH_2{\cdot}C_mH_{2m}{-}O{\cdot}CO{\cdot}R^2$; n = 1 or 2, m = 1, 2 or 3). Or coupling may precede *O*-acylation. Thus the monoazo compound *p*-nitroaniline → *m*-(bis-β-hydroxyethylamino)acetanilide is refluxed with acetic anhydride to give—



which dyes cellulose acetate scarlet. E.S.

Blue Monoazo Thiazole Disperse Dyes
ICI *BP* 856,898 (4 Sept 1958)

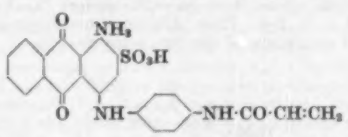
2-Amino-5-nitrothiazoles which may be substituted in the 4-position by Alk, CN, CF_3, phenyl, or substituted phenyl, are diazotised and coupled with *N*-epoxyalkyl derivatives of aniline to give the title dyes. Thus 2-amino-5-nitrothiazole → *N*-ethyl-*N*-βγ-epoxypropylaniline dyes cellulose acetate bright reddish blue. E.S.

Azo Acid Dyes having Acryloylamino Groups
BASF *BP* 858,183 (Germany 13 Dec 1957)

The title dyes are preferably monoazo compounds derived from a diazo component containing the group $-NH{\cdot}CO{\cdot}CH{:}CH_2$ and a coupling component containing a SO_3H group, and on wool, nylon, etc. are faster to wet treatments than similar dyes having a saturated acylamino group instead of the acryloylamino group. Thus the monoazo compound 4-(acryloylamino)aniline → Schaeffer acid dyes wool bright red from a weakly acid bath. E.S.

Azo and Anthraquinone Compounds Containing Acryloylamino Groups— Reactive Dyes for Cellulose
BASF *BP* 858,184 (Germany 14 Dec 1957)

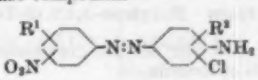
Suitable water-soluble dyes containing acryloylamino groups may be padded on cotton and fixed by an alkaline aftertreatment, or salt and alkali may be added to the dyebath and fixation effected by raising the temperature. Thus the monoazo compound 4-(acryloylamino)aniline → 3-methyl-1-*m*-sulphophenyl-5-pyrazolone is a yellow, and—



is a blue. E.S.

Aminomonoazo Compounds— Azoic Diazo Components
FH *BP* 856,929 (Germany 9 Feb 1956)

Aminomonoazo compounds—



(R^1 = H, Hal, Alk or OAlk; R^2 = H, Alk or OAlk) used as azoic diazo components in the production of clarets, garnets, etc. by coupling with arylides of 3,2-hydroxynaphthoic acid, give more stable diazo compounds and stronger dyeings and prints than widely used compounds, e.g. 4-amino-3,2′-dimethylazobenzene. Thus cotton is impregnated with an alkaline solution of the 4-chloro-2,5-dimethoxyanilide of 3,2-hydroxynaphthoic acid, and then developed in a diazo solution prepared from 4-amino-3-chloro-3′-nitroazobenzene to give a claret. E.S.

Acetoacetamide Derivatives of Phthalocyanines— Coupling Components for Azoic Greens
FBy *BP* 858,070 (Germany 27 Mar 1957)

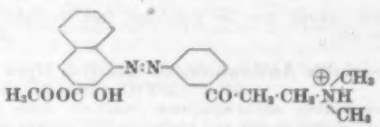
Phthalocyanines containing 1–8 aminomethyl groups, prepared as described in *BP* 717,137 and 724,212 (J.S.D.C., **71** (1955) 112, 263), are treated with diketen to give acetoacetamide derivatives—

$$Pc(CH_2{\cdot}NH{\cdot}CO{\cdot}CH_2{\cdot}CO{\cdot}CH_3)_n$$

(n = 1–8; Pc = phthalocyanine radical), which couple on the fibre with suitable diazo compounds to give greens. Thus octa-aminomethyl-copper phthalocyanine in fine aq. dispersion is stirred at room temperature with diketen to give the octa-acetoacetamide derivative. Cotton is impregnated with this compound, squeezed, dried, and developed in a diazo solution prepared from 2-amino-5-nitroanisole, to give a bright green. E.S.

Basic Dyes Containing Keto Groups
BASF *BP* 857,391 (Germany 23 May 1958)

Dyes containing a $-CO{\cdot}CH_3$ group are treated with formaldehyde, or compounds which yield formaldehyde, in presence of secondary or tertiary amines, so that the $-CO{\cdot}CH_3$ group becomes $-CO{\cdot}CH_2{\cdot}CH_2{\cdot}NR^1R^2$ (R^1, R^2 = Alk, or with N form a ring). Quaternary salts may be formed, and the products dye cellulose, cellulose acetates, nylon, polyester, and polyacrylonitrile fibres. Dyes of various chemical classes may be used, e.g. anthraquinone compounds, and particularly monoazo dyes derived from β-naphthol or 3,2-hydroxynaphthoic acid. Thus the monoazo compound *m*-aminoacetophenone → methyl 3-hydroxy-2-naphthoate is heated, in presence of acetic acid and a little HCl, with paraformaldehyde and dimethylamine to give—



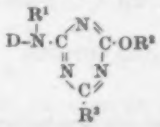
which dyes cotton, nylon, and cellulose acetate scarlet red. E.S.

Metal Compounds of Disazo Pyrazolone Dyes— Solvent-soluble Colorants
FH *BP* 857,584 (Germany 31 Mar 1956)

Cu, Ni, Co or Zn compounds of disazo compounds obtained by coupling diazotised 4-aminoazobenzenes (which may carry COOH or CH_3 groups) with mono- or di-sulphonic acids of 1-phenyl-5-pyrazolone-3-carboxylic acid are soluble in lacquers, and are faster to light than the salts of these metal-free disazo compounds with organic bases described in *BP* 814,605 (J.S.D.C., **75** (1959) 471). As these dyes contain only one OH group in *o*-position to an azo link it is doubtful if the metal compounds formed are complexes of the type given by *oo*′-dihydroxyazo compounds. Thus the disazo compound 4-aminoazobenzene→1-*o*-sulphophenyl-5-pyrazolone-3-carboxylic acid is mixed at 60°C with aq. $CuSO_4$, and stirring continued 8–10 h at room temperature, to give a reddish orange for nitrocellulose and similar lacquers. E.S.

Reactive Dyes for Cellulosic Fibres
By *BP* 857,186 (Germany 2 Aug 1957)

Fast-to-washing reactive dyes for cellulose are—

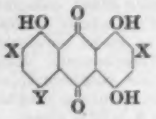


(D = dye radical; R^1 = H or Alk; R^2 = Ar; R^3 = OH or aryloxy). Fixation on the fibre, by heating in alkaline medium, is stated to occur in the 3-position, or if R^3 = aryloxy, in the 5-position of the triazine ring (with elimination of one or both aromatic OH groups). The dyes are prepared, e.g. by condensing 1 mol. cyanuric chloride with 2 mol. phenol or naphthol (I), pref. containing SO_3H groups, and having no dye character, and finally condensing with 1 mol. of a dye (II) containing $-NH_2$ or –NHAlk groups. Thus, where I is 1-hydroxy-2-nitrobenzene-4-sulphonic acid and II is the acid-coupled azo dye from 1-amino-4-acetamidobenzene-2-sulphonic acid → 2-amino-8-hydroxynaphthalene-6-sulphonic acid, hydrolysed, cellulosic fibres are coloured reds of good washing fastness. A.T.P.

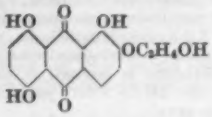
α-Hydroxy-β-Alkoxy- or Aryloxyanthraquinones—Disperse Dyes for Polyester Fibres

Ciba *BP* 856,553 (Switzerland 17 July 1956)

The title compounds—



(Y = H or OH, at least one X = subst. or unsubst. phenoxy or alkoxy, the other may be H) are orange to red dyes for synthetic-polymer fibres, especially polyester fibre, and have good affinity, light fastness, and wool reserve. Thus, 1,2,5,8-tetrahydroxyanthraquinone (12) is heated at 140°C with ethylene chlorohydrin (18), Cu acetate (0·25), and Cu bronze (0·25) in nitrobenzene (100) until a test portion becomes reddish violet in $NaOH/C_2H_5OH$. The mass is steam-distilled and filtered to give —

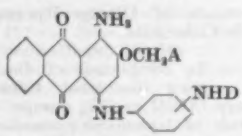


a reddish orange dye for Terylene, fast to light and to sublimation. A.T.P.

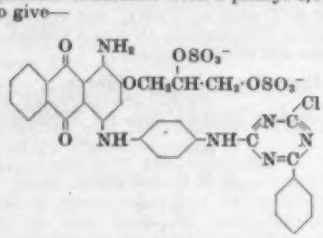
Water-soluble Anthraquinone Reactive Dyes

BASF *BP* 859,283 (Germany 31 Aug 1957)

Water-soluble anthraquinone reactive dyes contain reactive halogen atoms and hydroxyalkyl groups esterified with H_2SO_4 or aryl carboxylic acids containing $\geqslant 1$ SO_3H group. Thus, they may be—



(A = subst. or unsubst. aliphatic radical, carrying $\leqslant 1$ OH esterified as above, D = heterocyclic radical containing $\leqslant 1$ grouping $-C(Hal)=N-$ in the heterocyclic ring, or D = acyl radical of an aliphatic α- or β-halogeno-carboxylic acid. Thus, 1-amino-2-βγ-dihydroxypropoxy-4-*p*-aminoanilinoanthraquinone is esterified with H_2SO_4 and the product condensed with 2-phenyl-4,6-dichloro-s-triazine to give—



which dyes wool and polycaprolactam blue. A.T.P.

Acedianthrone Vat Dyes

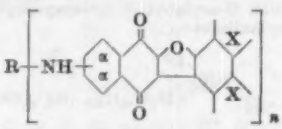
Ciba *BP* 858,586 (Switzerland 4 Oct 1956)

1 Mol. dihalogenoanthraquinone or dihalogenobenzanthrone reacts at 100–350°C in a high-boiling solvent in presence of an acid-binding agent and Cu catalyst with 2 mol. monoaminoacedianthrone (I) (from the mononitration and reduction of acedianthrone) or 1 mol. I and 1 mol. monoiminoanthraquinone, and the product is optionally carbazolised to give brown vat dyes, especially suitable for printing. Thus, dyes from 2,7-dichloro- or 1,5-dichloroanthraquinone and I are reddish brown and yellow-brown respectively. A.T.P.

Heterocyclic Vat Dyes

General Aniline *BP* 856,803 (U.S.A. 30 Dec 1955)

Multi-component mixtures of—

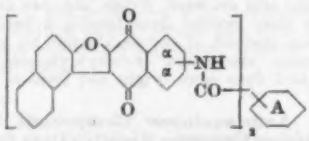


(one adjacent pair of X valencies is joined to H atoms, the other pair to a group—

(*n* = 2–3; R = bi- or tri-valent benzene or diphenyl radical, lower Alk or hydroxyalk; NH groups are in either α position) are bright vat dyes of excellent fastness to washing, light, and chlorine. They are obtained by condensing 1 mole of RBr_n with *n* mole of a mixture of requisite intermediates of *BP* 835,908 (J.S.D.C., **76** (1960) 510). Thus, *p*-dibromobenzene (1·7) is condensed at 200–205°C for 20 h in nitrobenzene in presence of sodium acetate and Cu_2Cl_2 with the mixture of intermediates (4·5) of Example 1 of *BP* 835,908 to give a mixture dyeing cotton greys of good fastness to chlorine, light, and washing.

BP 856,804 (U.S.A. 30 Dec 1955)

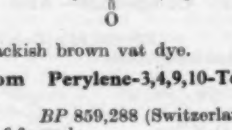
The intermediates of *BP* 835,908 are treated with aromatic or heterocyclic acylating agents derived from a mono- or di-carboxylic acid to give multi-component mixtures of heterocyclic vat dyes. Thus, from terephthaloyl chloride (1) and the intermediates of Example 2 of *BP* 835,908 (3·1), is obtained a bright orange vat dye, fast to light, chlorine, and washing—



(NH groups in one or other α-position, CO groups in *p*-positions in A).

BP 856,805 (U.S.A. 30 Dec 1955)

Similar dyes result from the reaction of 2, 3, or 4 mol. of the aforementioned intermediates with nuclearly di-, tri-, or tetra-halogeno-substituted vattable polycyclic ketones (I). They have the same structure as the dyes of *BP* 856,803, where R = an *ortho*-nuclear bonded residue of I and *n* = 2–4. Thus, dibromodibenzopyrenequinone (1·2) and a mixture of the two isomers (1·6)—

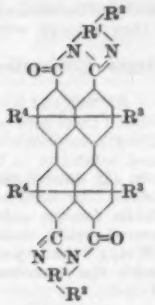


gives a fast blackish brown vat dye. A.T.P.

Pigments from Perylene-3,4,9,10-Tetracarboxylic Anhydride

S *BP* 859,288 (Switzerland 29 Oct 1957)

Compounds of formula—



(and its isomer) (R^1 = radical of an *ortho*- or *peri*-arylene, R^2 = H, Hal, Alk, Alkoxy, R^3 and R^4 = H or Hal) are suitable for colouring nitrocellulose lacquers, printing inks, plastics, and for mass-coloration of regenerated cellulose, cellulose esters and ethers, and synthetic polymers with good fastness to light, washing, migration in polyvinyl chloride, top coating finishes in lacquers, overprinting in inks, heat in all plastics (especially in molten polyamides and polyesters) and chlorine and peroxide bleaching. They are obtained by condensing perylene-3,4,9,10-tetracarboxylic anhydride (I) or its halogen derivatives with diamines, and optionally halogenating the product. Thus are obtained pigments from I and 1,2-diaminobenzene (II) (violet on PVC), 1,8-diaminonaphthalene (reddish blue) and bromo-I and II (reddish violet).

A.T.P.

Non-staining Triarylmethane Colour Base and Printing Therewith

C. Davies *USP* 2,927,040–1 (13 Aug 1957)

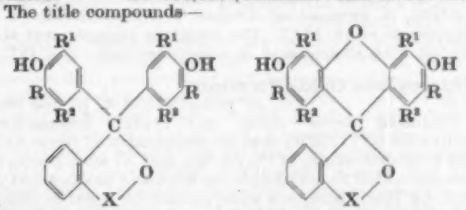
Introduction of nitro groups into triphenylmethane dye bases lowers their dissociation constants to a level where the resultant dye is too weakly basic to dye the skin yet is still sufficiently acidic to yield intensely coloured salts with suitable acids, including paper coating acid clays and acid silicas. Thus Dinitro Crystal Violet Carbinol Base and Dinitro Malachite Green Carbinol Base yield strong yellow solutions in organic solvents which can be handled without leaving intense violet or green stains on the skin. Used as secondary fluids they give intense markings on suitable secondary surfaces. Thus a 3% solution in toluene of 2,2′-dinitro-4,4′,4″-tris(dimethylamino)-triphenylcarbinol (Dinitro Crystal Violet Carbinol Base) gives an intense dark violet, almost blue-black, coloration to paper coated with unactivated silica.

C.O.C.

Phthalein and Fluorescein Derivatives

J. Korbl *BP* 858,019 (Czechoslovakia 16 Oct 1956)

The title compounds—

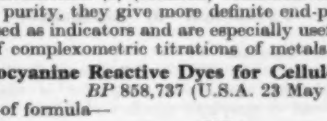


are obtained by condensing fluorescein or phthalein derivatives of the above structure (R = H), with iminodiacetic acid and HCHO in HCOOH or CH_3COOH, in presence of sufficient univalent cations to dissolve some or all of at least one of the insoluble components. When isolated by precipitation with a solvent, e.g. CH_3OH, they are obtained in a much higher purity than similar products from conventional (I) (alkaline) condensations. Because of their purity, they give more definite end-points than I when used as indicators and are especially useful in a wide range of complexometric titrations of metals. A.T.P.

Phthalocyanine Reactive Dyes for Cellulose

BASF *BP* 858,737 (U.S.A. 23 May 1958)

Dyes of formula—



(A = phthalocyanine residue; B = aromatic radical containing a mono- or bi-cyclic nucleus; m = 1–8; n = 0–4; Z = $-C(R^1)=C(R^2R^3)$; R^1 and R^2 = H, Alk, hydroxyalkyl or Hal; R^3 = H, Alk, hydroxyalkyl, Hal or carboxyl) are applied to cellulosic fibres in conjunction with an acid-binding agent and (optional) finally heated at 70–150°C for a short time to give blue and green dyeings of excellent fastness to washing, rubbing, and light. They may be obtained by treating phthalocyanine compounds of *BP* 827,569 (J.S.D.C., **76** (1960) 318) with acryloylating agents (cf. *BP* 853,364 (J.S.D.C., **77** (Feb 1961) 79). Thus, regenerated cellulose is padded with an aqueous solution containing the dye (20) from acrylic acid chloride and the reaction product of 1 mol. pentakischloromethylmonochlorocopper phthalocyanine and > 5 mol. 1-hydroxy-2-aminobenzene-4-sulphonic acid, Na_2CO_3 (25) and Na_2SO_4 (20), and the fibre is dried and steamed for 6 min at 102°C to give a turquoise blue of excellent light fastness.

A.T.P.

Phthalocyanine and Anthraquinone Pigments of Fine Particle Size

Ciba *BP* 858,068 (Switzerland 20 July 1956)

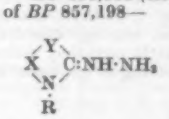
Pigments of high colour strength and soft grain, readily dispersible in a wide variety of substrates, are obtained when crude fully oxidised phthalocyanine or anthraquinone pigments are heated for < 5 h at 100–300°C in aqueous medium. The amount of aqueous medium must at least be sufficient to surround each pigment particle by an aqueous phase.

C.O.C.

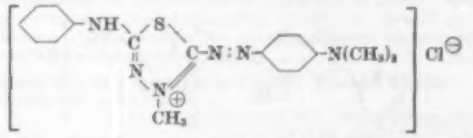
Diazapolymethin Basic Dyes for Polyacrylonitrile Fibres

BASF *BP* 858,181 (Germany 26 Feb 1957)

The hydrazones of *BP* 857,198—



(R = Alk or Ar; Y = S or O, or NH, NAlk, NAr, or $N(NH_2)$; X = divalent azomethin radical subst. on the C atom) are condensed in presence of oxidising agents with azo coupling components, especially arylamines or indole derivatives, to give the title dyes. Thus 5-anilino-3-methyl-1,3,4-thiadiazolone(2)-hydrazone hydrochloride in ethanol is stirred with *NN*-dimethylaniline hydrochloride. Aq. $FeCl_3$ is run in and at the same time an aq. solution of NaOH and sodium acetate, to give—



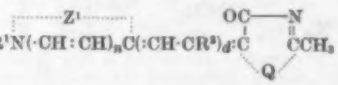
which dyes polyacrylonitrile fibres blue.

E.S.

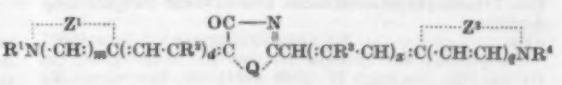
Merocyanines and Planar Undissociated Cyanines

Eastman Kodak Co. *USP* 2,927,026 (30 Aug 1956)

Preparation of dyes of formula—



(R^1 = C_mH_{2m+1} (m = 1–4); R^2 = H, Alk or Ar; n = 0 or 1; d = 0, 1 or 2 (when d = 2, R^3 = H); Q = O, divalent S, NR^3 (R^3 = Alk or Ar), or atoms to complete a 6-membered ring; Z^1 = atoms to complete a 5- or 6-membered ring), e.g. 5-(3-ethyl-2(3*H*)-benzothiazolyidene)-2-methylthiazolin-4-one, is described. Their methyl group can react with suitable compounds to yield planar undissociated cyanine dyes of formula—

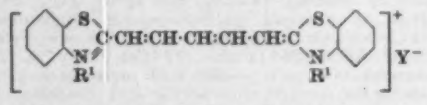


(x = 0, 1 or 2; R^3 = H, Alk or Ar; R^4 = C_mH_{2m+1} (m = 1–4); Z^2 = atoms to complete a 5- or 6-membered ring;

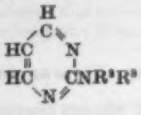
q = 0 or 1), e.g. 1-phenyl-5-[1,3,3-trimethyl-2(3*H*)-indolylidene)ethylidene] - 2 - [3 - (1,3,3 - trimethyl - 2(3*H*) - indolylidene)propenyl] - 4,6(1*H*,5*H*) - pyrimidinedione. C.O.C.

Pentamethincyanines
Ilford *BP* 859,452 (14 May 1958)

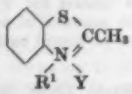
Pentamethincyanine dyes of formula—



(R^1 = Alk, Ar or aralkyl; Y = acid radical) are obtained by treating 1 mol. of a compound of formula—



(R^2 = H, Alk, Ar, aralkyl or carboxylic acyl; R^3 = H, Alk, Ar or aralkyl) with two mol. of a compound of formula—



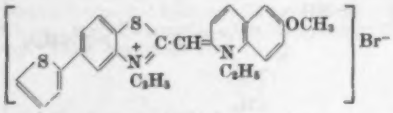
in presence of an acid-binding agent. Thus the blue bis-2-(3-ethylbenzothiazole)pentamethincyanine iodide is obtained by fusing 2-methylbenzothiazole with ethyl toluene *p*-sulphonate, cooling, dissolving in 2-acetamino pyrimidine and a little pyridine, refluxing, pouring into hot aqueous NaI, cooling in ice water and filtering off the precipitated dye. C.O.C.

New Cyanine Dye
Kodak *BP* 852,955 (U.S.A. 25 June 1956)

The dye, 4-[(2-ethyl-1(2H)-s-triazolo[3,4-b]benzothiazolylidene)ethylidene] - 3 - methyl - 1 *p* - sulphophenyl - 5-pyrazolone is especially useful for extending the spectrasensitivity of photographic emulsions. C.O.C.

Cyanine Dyes
Agfa *BP* 855,686 (Germany 28 Mar 1957)

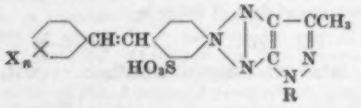
Cyanine dyes derived from benzothiazoles and substituted in the 5- or 6-position by a thiophen radical, e.g.—



have powerful photosensitising properties. C.O.C.

Fluorescent Brightening Agents
HWL *BP* 855,784 (2 Sept 1957)

Compounds of formula—



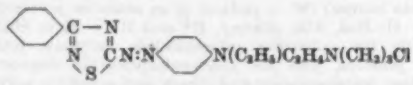
(X = H or a non-chromophoric and non-auxochromic atom. or group; n = 1–5; R = H or subst. or unsubst. hydrocarbon) show blue fluorescence in ultraviolet radiation. They are fast to bleaching agents both in solution and on the fibre. Thus 2-(4″-styryl-3″-sulphophenyl) (1′-phenyl-3′-methylpyrazolo-4′,5′,4,5)-1,2,3-triazole applied to cotton in a detergent solution yields a brilliant white. C.O.C.

Bis-Triazinylaminostilbene Fluorescent Brightening Agents
FBy *BP* 855,480 (Germany 6 June 1957)

Compounds of formula similar to those given in *BP* 847,725 (J.S.D.C., **77** (Feb 1961) 82), but where Na may be replaced by another cation or H, show strong bluish fluorescence in ultraviolet radiation. They may be applied to a wide variety of substrates but are particularly useful for cotton, as they exhaust very evenly on to it. C.O.C.

Basic Dyes for Mass Coloration of Acrylonitrile Polymers
FBy *BP* 855,153 (Germany 18 Oct 1957)

Basic dyes which have been precipitated from aqueous solution, e.g. by adding a sulphonic acid to the solution, show greatly increased solubility in the solvents used for acrylonitrile polymers, e.g. dimethylformamide, compared with the original dyes. In addition they contain no entrained water-soluble foreign salts. Yarns produced from polymers coloured with these dyes have better fastness to cross-dyeing than yarns produced from polymers treated with the corresponding water-soluble dyes. Thus the red dye



is precipitated with Na propane-1,3-disulphonate. C.O.C.

Stains for Cellulose, Paper, Wood, etc.
Commonwealth Engineering Co. *USP* 2,921,829 (25 June 1956)

Stains which are non-grain raising and fast to light are obtained by using an alkaline degradation product of a diazotised amine as the colorant. Thus aniline is dissolved in water containing 3 equivalents of HCl at 0–5°C and aqueous $NaNO_2$ slowly added until excess HNO_2 (as shown by starch–iodine) is present. The solution is cooled to 10–20°C and poured into excess of 10–30% aq. NaOH, acidified with HCl and the brown product extracted with toluene to form a staining composition. C.O.C.

Orange to Red Inorganic Pigments containing Cadmium and Mercury
James M. Brown *BP* 856,428 (16 Mar 1956)

Calcining a mixture of Hg and Cd compounds yields orange to red pigments of excellent fastness to heat and light. Thus HgO (17), S (3) and CdS (80) heated in a closed crucible for 8 h at 520°C yield a red pigment. C.O.C.

Coated Calcium Carbonate Pigments (C.I. Pigment White 18)
Wyandotte Chemicals Corpn. *USP* 2,927,091 (1 Sept 1953)

A pigment of excellent quality and readily dispersible in rubber mixes is obtained by coating finely powdered $CaCO_3$ with both (*a*) a C_{10-18} primary *n*-alkylamine or rosin amine and (*b*) a C_{10-20} fatty acid or rosin acid. Thus $CaCO_3$ was precipitated by mixing aqueous solutions of $CaCl_2$ and $NaHCO_3$ in presence of Armeen T (hydrochloride of an alkylamine of 16–18 C). The resultant pigment was then treated with stearic acid in a hammer mill. C.O.C.

Hydrous Iron Oxide Pigments
DuP *BP* 858,072 (U.S.A. 15 Apr 1957)

Hydrated ferric oxide (C.I. 77492), ferrous-ferric hydroxide (C.I. 77499) and co-precipitates of ferric oxide and hydrated oxides of Cr, Al, Mn, and Ni after precipitation are added to a hydrophobic aliphatic monocarboxylic acid, e.g. lauric acid, in a water-immiscible volatile organic solvent for the acid, and the mixture is agitated. This results in much of the acid becoming bound to the surface of the pigment particles by salt linkages. The product is hydrophobic and of particle size below the visible light-scattering level. Paints, etc. containing this pigment have superior transparency, brilliance, two-tone effect and depth of mass tone compared with those paints with ordinary hydrated iron oxide pigments. C.O.C.

Electrolytic Production of Manganese Dioxide (C.I. 77728)
Cartoucherie Française *BP* 859,723 (France 28 July 1958)

A solution of manganese sulphate is electrolysed between a lead or graphite anode and a lead cathode in intimate contact with natural MnO_2, e.g. coated into the cathode. This method needs much less electrical power than previous methods and prevents release of hydrogen at the cathode. C.O.C.

Carbon Black (C.I. Pigment Black 6 and 7)
Phillips Petroleum Co.
BP 854,750 (U.S.A. 29 June 1956)

Liquid coolant is introduced directly into the reaction mixture in the furnace proper, and not at a point downstream from it. The properties of the resulting pigment vary substantially, according to the point within the furnace at which the initial quenching is effected. C.O.C.

Silica (C.I. Pigment White 27) of Fine Particle Size
Henkel & Cie. *BP* 859,271 (Germany 30 Aug 1958)

A mixture of quartz sand, carbon and a polyvalent metal which will reduce SiO_2 is heated at < 1400°C and the resulting SiO oxidised to SiO_2. The polyvalent metal, e.g. Al dust, is present in the proportion of 0·02–0·2 mol. per mol. SiO_2. Processing to SiO occurs at a satisfactory rate and yield at < 1600°C. C.O.C.

Silica (C.I. Pigment White 27)
Columbia-Southern Chemical Corpn.
USP 2,924,510 (24 Dec 1956)

A slurry of silica is obtained by treating aqueous Na silicate with acid. This is then treated with a little dialkyldimethylammonium chloride (Alk of 8–18 C), diluted, washed with hot water and allowed to settle in conventional thickening equipment. The clear liquor is withdrawn and the thickened slurry filtered to yield very pure pigmentary silica. C.O.C.

1960 Reviewed. Technical Developments in the Pigment, Paint, and Printing Ink Industries (V this page)
Organic Protective Coatings (V this page)
Mass Coloration of Cellulose Acetate and Synthetic Polymers (VI p. 135)
Red Mass-coloured Cellulose Acetates (VI p. 135)
Reactive Dyes for Cellulosic Fibres and their Application (VIII p. 138)
Sensitisation of Photoconductivity in Glass by Dyes (XIV p. 145)
Calcein Blue—a Metalfluorochromic Indicator for Chelatometric Titrations (XIV p. 145)

V—PAINTS; ENAMELS; INKS

1960 Reviewed. Technical Developments in the Pigment, Paint, and Printing Ink Industries
Paint, Oil & Col. J., 139 (6 Jan 1961) 49–54

Brief surveys of the literature under the following headings: pigments (24 references), paints (20), varnishes (6), oils (10), resins (25), plant and equipment (6), printing inks (7), analysis and testing (6), corrosion and weathering (7). C.O.C.

Philosophy of Coatings
H. F. Payne *Off. Dig. Fed. Soc. Paint Technology*, 32 (Nov 1960) 1371–1434

Binding forces in matter are explained and discussed under the following headings: electronic forces; metallic, electrovalent, and covalent structures; co-ordinate covalent bonding; polarity and dipole moment; van der Waals forces; permanent and induced dipole forces; London force of attraction; the H bond; atomic structure; electrons in chemical reactions; energy levels in atomic structures; electron orbits; angular momentum; atomic and ionic dimensions; ionisation potential; electro-positive and -negative elements; energy values for single bonds; bond formation; metallic, electrovalent, and covalent bonds; the H_2O molecule; covalent double and triple C bonds; resonance. The application of these basic concepts is discussed under these headings: C cpd.; hydrides of C and other elements; cpd. of B, Si, F, and P; inorganic coatings. Further developments may be expected along four lines: (*a*) improvement of organic binders through better control of their mol. structures, e.g. by graft, block and stereospecific polymerisation; (*b*) improvement of resistance of organic binders to heat, short-wave radiation, and chemicals, by incorporation of metals such as Al, Ge, Sn, Pb, and Fe in their mol. structures; (*c*) entirely new performance from inorganic binders designed to have toughness in addition to resistance to heat and radiation, e.g. by chemisorption on the substrate to enhance adhesion; and (*d*) development of new methods of application and curing of coatings. Greater stability may be secured by the radiation cross-linking of non-reactive binders, flame-curing may be used to remove polar groups from the surface of coatings to improve resistance to various agencies, and coatings may be "annealed" to relieve stresses produced during film formation. J.W.D.

Molybdate Pigments—New Corrosion Inhibitors
H. O. Schoen and B. G. Brand
Off. Dig. Fed. Soc. Paint Technology, 32 (Nov 1960) 1522–1543

The corrosion-inhibiting properties of Ca and Zn molybdates, applied to steel panels, were studied by accelerated exposure and exposure on two outdoor sites. Both equalled or exceeded red lead in protective power and are, moreover, non-toxic. When used as primers alone the molybdates were rated in the following order of effectiveness: Ca, normal Zn molybdate, Zn polymolybdate; when covered with a topcoat, however, the Ca cpd. was the least effective. With certain exceptions the molybdates were more efficient when extended with $CaCO_3$ (more effective than talc). Fine-particle molybdates were more effective than coarse. Linseed oil is a better vehicle for these cpd. than is alkyd resin. Increasing the Mo/Zn ratio from 1 to 1·4 reduced the corrosion resistance. J.W.D.

Organic Protective Coatings
A. T. S. Rudram
Chem. & Ind., No. 53 (31 Dec 1960) 1608–1616

Surface coatings are discussed, with special reference to the prevention of metallic corrosion, under the following headings: condition of the metal surface; conditions of exposure; application requirements; selection of the paint system; effect of pigments used in coatings; protection by chemical inhibition; inhibition by chrome and lead pigments; use of alkyds, tar and bitumen paints, treated and synthetic rubbers, dispersion and fusion coatings, and coatings formed by chemical reaction, e.g. epoxy resins. J.W.D.

Pigmented Coatings for Glass Fibre
Northwestern Society for Paint Technology
Off. Dig. Fed. Soc. Paint Technology, 32 (Nov 1960) 1449–1462

Glass-fibre laminated panels were treated with different finishes—vinyl-alkyd, epoxidised oil lacquer, amine-cured epoxy, polyamide–epoxy, oil-modified polyurethane, two-package polyurethane, styrenated alkyd, and acrylic lacquer. The finished panels were tested for adhesion (both initial and after Weather-Ometer and salt-spray exposure), abrasion resistance, Weather-Ometer exposure, salt-spray exposure, and resistance to chemicals. Further tests were made with five of these systems, together with two methods of fibre-surface preparation; in a third series the surfaces were not prepared at all. To obtain good adhesion it is essential to remove release agents and peroxide residues before coating. J.W.D.

Dispersion of Pigments in Highly Viscous Media
Translated by A. Fuerst
Off. Dig. Fed. Soc. Paint Technology, 32 (Oct 1960) 1313–1324

Translation from the Russian of a paper by I. M. Alekseev (*Khimicheskaya Nauka: Promyshlennost'*, 4 (May–June 1959) 365–371). The relation between pigment particle size and the properties of paint films is briefly outlined. Pigment dispersion is discussed under several headings: fine grinding of pigments on jet mills (micronisers), methods of dispersion of pigments in binders, processing of highly viscous coloured pastes (a two-roll rubber mill is preferred), influence of dispersing agents, composition of dry-milled pastes (chip compositions), and the production of high-quality nitrocellulose enamels—enamels produced from these chip compositions are claimed to have properties superior to those of enamels produced by other means. J.W.D.

Microrheology of Dispersing Pigments in Non-aqueous Media by Ball-milling
L. Dintenfass *Kolloid-Z.*, 170 (1960) 1–19 (*in English*)
Chem. Abs., 54 (10 Nov 1960) 23359

Various pigments were ball-milled with solvents, resins, and wetting agents, and the rheological properties and behaviour of the suspensions on dilution were determined. The influence on grinding efficiency of pigment and resin concentration, size of mill (0·5–2·0 gal), time of grinding, and nature of the medium were studied. Data on adsorption and a classification of rheological types previously

published (*Chem. Abs.*, **53** (1959) 2733; **54** (1960) 14717) were used to explain the results and establish generalisations helpful in making paints and inks. The polar groups of dispersing agents (e.g. resins, grinding assistants, driers) should be matched to the nature of the active areas on the pigments. If different types of active area are present, two or more agents with different polar groups are desirable. Alkyd resins are especially effective. Adsorption by the pigment of their fractions of lower mol. wt. tends to "upgrade" resins, but the solvent strength must be carefully chosen to permit adsorption while preventing precipitation. Diluents should be more powerful solvents and of lower mol. wt. than the original grinding medium. Soaps having no drying properties may increase drying by displacing adsorbed drier. Unless thixotropic alkyds are used, Newtonian characteristics are desirable for both solid and liquid phases. C.O.C.

Pigment Distribution and Covering Property
A. Goeb *Farbenchemiker*, **61** (12) (1959) 7–12
Chem. Abs., **54** (10 Nov 1960) 23359

An illustrated explanation of transparency measurement with a wet film to determine covering power. Data are given based on experiments with different samples of White Lead (C.I. Pigment White 1) of the relations between particle size, pigment concentration, n of the pigment, time of grinding, and velocity of sedimentation, on the one hand, and covering power on the other. C.O.C.

Lead Benzoate as a Paint Preservative
M. Nemec *Korose a ochrana materialu*, (1960) 15
Chem. Abs., **54** (10 Nov 1960) 23357

Addition of 2–3% Pb benzoate to rubber-based and similar-base coating compositions much increases their life after application. C.O.C.

Flotation and Flooding in Paints
A. B. Lock
J. Oil & Col. Chem. Assocn., **43** (Dec 1960) 859–870

Existing theories on flotation and flooding in paints are compared and discussed, and the important rôle of the vehicle is stressed. Flotation and flooding behaviour of TiO_2 with Synthetic Yellow Oxide, Lamp Black (C.I. Pigment Black 6), Phthalocyanine Blue (C.I. Pigment Blue 15), and Prussian Blue (C.I. Pigment Blue 27) in 5 alkyds of differing acid and OH values are studied. Several test methods are used and explained, and their relation to practical application tests is shown. It is difficult to assess and even to differentiate between flooding and flotation; this is discussed. Variations in acid and OH value differ in their effect on all the pigmentations tested. J.W.D.

Spectral Reflectivities of White Paints
V. R. Ivanuski and E. H. Tomkins
J. Oil & Col. Chem. Assocn., **43** (Dec 1960) 833–841

Spectral reflectivity data in the visible and near-infrared are given for white paint coatings. A small but significant absorption in the violet region of the visible spectrum is associated with poor pigment dispersion and with high porosity. Best wide-spectrum reflection is given by blends of MgO with bleached diatomaceous earth; overcoatings of MgO on rutile or anatase titania, however, do not increase significantly the near-ultraviolet reflectivity when coating thicknesses are reasonable. Pigmented ceramic coatings are among the best available reflectors for the near-infrared; in this connection, further attention might profitably be paid to silicon carbide and boron nitride. J.W.D.

Zinc Pigments in Exterior House Paints
Off. Dig. Fed. Soc. Paint Technology, **32** (Oct 1960) 1289–1312

Symposium presented by a panel representing the American Zinc Institute. In an introduction the research and test facilities available, on a co-operative basis, within the Institute are outlined. There follows a discussion in which a wide variety of questions relating to zinc pigments and their applications are posed to and answered by members of the panel. J.W.D.

PATENTS

N-Aliphatic-β-aminobutyrates as Stabilisers for Emulsion Paints against Freezing and Thawing
Armour & Co. *BP* 857,944 (U.S.A. 5 Apr 1957)

Addition of a compound of formula—

$$RNHCH(CH_3)CH_2COOM$$

(R = aliphatic hydrocarbon of 8–12 C; M = Na or K), e.g. Na *N*-coco-β-aminobutyrate, stabilises emulsion paints against repeated freezing and thawing. C.O.C.

Thixotropic Emulsion Paints
British Oxygen Research & Development *BP* 857,738 (13 Nov 1958)

Paint based on an aqueous emulsion of a polyalkyl acrylate and/or polyalkylmethacrylate is rendered thixotropic by addition of 0·05–6·0% by weight of a compound of formula—

$$nSiO_2 \cdot M_2O$$

(M = alkali metal; n = 0·5–4·2) e.g. commercial sodium silicate of mean composition $3{\cdot}3SiO_2 \cdot Na_2O$. C.O.C.

Wrinkle Finish Composition
Union Carbide Corpn. *BP* 855,756 (U.S.A. 29 Apr 1958)

A hard, tough, scratch-resistant wrinkle finish is obtained by use of a plastisol consisting solely of a vinyl resin (5–80% by wt.) and a liquid plasticiser (95–20). Not less than 30% by wt. of the plasticiser is a liquid polymerisable ester of an αβ-ethylenically unsaturated monocarboxylic acid of 3–6 C with an alkylene glycol. To this plastisol is added as drier 0·01–10% (on wt. of the ester) of a polyvalent metal salt of a saturated monocarboxylic acid and any derived pigment. C.O.C.

Coating Composition yielding a Bead Surface Finish
Midland Chemical Corpn. *BP* 855,152 (30 Sept 1958)

Small beads or spheres of a heat-expandable resin are incorporated into a vehicle, e.g. of the "force dry" or baking type. Upon the resultant composition being applied and heated, the resin expands producing a coating composed of a multitude of closely packed expanded beads. C.O.C.

Fine Dispersions of Carbon Black (C.I. Pigment Black 6 and 7) in Polyethylene Compositions
Union Carbide Corpn. *BP* 856,314 (U.S.A. 27 Nov 1957)

Carbon Black (10–150 parts by wt.) is blended with polyethylene particles (100) and water (1–9 on wt. of pigment) until the polyethylene particles are coated with a coherent mixture of pigmented water. The blend is then worked under conditions of high shear until the polyethylene fluxes and the water is eliminated from the mixture leaving the pigment uniformly disposed throughout the fluxed polyethylene. This forms a master batch of pigmented polyethylene which is used, e.g. in a Banbury mixer, at fluxing temperature, to pigment additional polyethylene to a pigment content of 2–6%. The product is especially suitable for outdoor use as it is resistant to degradation and yields a smooth glossy coating. C.O.C.

Aqueous Polyvinyl Acetate Coating Compositions
ICI *BP* 854,346 (4 July 1958)

When the same water-soluble colloidal thickeners are added during polymerising and during compounding of vinyl acetate dispersions, the resulting composition yields coatings of improved gloss and resistance to wet rubbing. C.O.C.

Polyurethane Lacquers
ICI *BP* 853,384 (1 July 1957)

Premature curing of polyurethane coatings and lack of need to use organic solvents is achieved by successively applying an aqueous dispersion of a polyester or polyesteramide containing no soap or dispersing agent except alkali and then a polyisocyanate. C.O.C.

Quick-drying Vehicles for Printing Inks
Miehle–Goss–Dexter *BP* 852,944 (11 Feb 1959)

The vehicle consists essentially of a drying oil binder having a conjugated system of double bonds, e.g. bodied tung oil, a cross-linking agent, e.g. divinylbenzene, and an oil-soluble acyl halide photopolymerisation catalyst, e.g. cinnamoyl chloride. Inks based on such vehicle dry within a few seconds when exposed to ultraviolet radiation but nevertheless have a long life in storage before use. The catalyst does not cause livering of the vehicle and in many cases does not cause skinning. C.O.C.

Magnetic Lithographic Ink
A. B. Dick Co. *BP* 857,820 (U.S.A. 14 Oct 1958)

A magnetic lithographic ink comprises: lithographic varnish (20–60 by wt.), magnetic iron oxide (C.I. 77499) (20–75), toner (0–20), and surfactant (0–15). C.O.C.

Flame-retardant Coating Compositions

Pearl Varnish Co. *BP* 853,759 (29 May 1959)

A composition, especially suitable for application to fibre board, comprises an aqueous emulsion of a film-forming organic addition polymer, Na borate, a halogenated organic compound and leafing aluminium powder (C.I. Pigment Metal 1) with or without other inorganic pigments and/or extenders. The preferred aluminium powder is the water-dispersible Aqua Silver (English Metal Powder Co.). C.O.C.

Pigments from Perylene-3,4,9,10-Tetracarboxylic Anhydride (IV p. 128)

Non-staining Triarylmethane Colour Base and Printing Therewith (IV p. 129)

Hydrous Iron Oxide Pigments (IV p. 130)

White Pigments and White Paperboard (XI p. 143)

VI— FIBRES; YARNS; FABRICS

Microscopical Staining of Fibre Cross-sections with Procion Black HGS (C.I. Reactive Black 1)

K. Katō *J. Soc. Textile Cellulose Ind. Japan*, **16** (Oct 1960) 827–831

The procedure previously outlined (J.S.D.C., **75** (1959) 631; **76** (1960) 64) was re-examined. Effect of washing stained fibres with a 30% aqueous soln. of pyridine was used as a criterion of the extent of chemical bonding between the reactive dye and the fibre occurring during staining. It is concluded that the staining of cellulosic fibres and Vinylon is not chemical in nature, whereas staining of protein fibres is. The differential staining observed on viscose rayon is therefore caused in the same way as with other types of dye. Six photographs show, respectively: (1) staining of viscose rayon and casein at pH 3 and 10, and effect of washing with water and 30% aqueous pyridine, (2) staining with hydrolysed Procion Black HGS, (3) effect of pH on skin staining by Azine Brilliant Blue, (4) residual staining on viscose rayon after washing, (5) effect of acetalisation on staining of polyvinyl alcohol, (6) effect of acetylation on staining of casein fibre. M.T.

Affinity for Cellulose of Polyenedicarboxylic Acids of the Crocetin Type and of Quaternary Ammonium Compounds

R. Wirz and H. Zollinger

Helv. chim. Acta., **43** (Oct 1960) 1738–1745

Eight polyenedicarboxylic acids were synthesised and the affinity of each for cellulose determined. From results obtained for the six derivatives possessing only double bonds, it is concluded that substantivity increases with increasing chain length and number of conjugated double bonds (3 to 9). It is suggested that van der Waals forces are mainly responsible for the substantivity, although hydrogen bonding cannot be excluded as a possible means of attachment. The substantivity of two derivatives having a central triple bond was found to be greater than that of the analogous compounds with a central double bond, despite the departure from complete linearity due to the triple bond. The substantivity of diphenyl-4,4′-bis-trimethylammonium hydroxide (bis-quaternised benzidine), and the corresponding *m*-tolidine derivative was determined, in the presence of salt, at various temperatures. It was concluded that the substantivity in these cases is due exclusively to van der Waals forces. The effect of salt on the substantivity is discussed, the increase in substantivity of the benzidine derivative with rise in temperature being considered to be due to aggregation taking place preferentially at lower temperatures. I.G.L.

A Study of "Static" in Relation to the Soiling of Garments in Wear

J. Lord and W. H. Rees

J. Textile Inst., **51** (Oct 1960) T 419–T 428

Details are given of a study of the generation of electrostatic charges on the collars and cuffs of cotton, Terylene and Orlon shirts and on the skirt portions of nylon, Terylene and Tricel underwear during wear. By comparing parts of the garments treated with anti-static agent to eliminate electrostatic charges with untreated parts of the garments, the effect of such charges on the soiling of garments in wear was examined. No significant difference in the level of soiling was found between treated and untreated parts of any of the shirts, so that there is no obvious soiling effect which could be attributed to "static". On the other hand, the untreated parts of the skirts of the underwear soiled and yellowed to a greater extent than the treated parts. W.G.C.

Sorption of Water by Cellulose and Eight other Textile Polymers

R. Jeffries

I— Sorption of Water by Celluloses below 100°C

J. Textile Inst., **51** (Sept 1960) T 339–T 374

Detailed results are presented on the equilibrium absorption and desorption of water vapour by twelve celluloses in the range 0–100% r.h. and 30–90°C. As the celluloses had been previously stabilised by repeated sorption treatments and were non-standard materials, the results are of theoretical rather than practical interest.

II— Sorption of Water Vapour Below 100°C by Textile Polymers other than Cellulose

Ibid., **51** (Oct 1960) T 399–T 418

Detailed results are presented on the absorption and desorption of water vapour (0–100% r.h., 30–90°C) by cellulose triacetate, secondary cellulose acetate, ethylcellulose, drawn nylon 6.6 yarn, Terylene, Orlon, wool and Fibrolane. Previous work on these polymers is compared with the present results.

III— Sorption of Water Vapour by Textile Polymers at 120°C and 150°C

Ibid., **51** (Nov 1960) T 441–T 457

The last paper of this series presents results on the absorption of water vapour (0–100% r.h.) at 120°C and 150°C. Desorption measurements were also made on cotton, viscose rayon, and secondary cellulose acetate. W.G.C.

New Cellulosic Fibres

I. H. Welch and W. S. Sollenberger

Amer. Dyestuff Rep., **49** (19 Sept 1960) P 695–P 700

Three new rayon staple fibres and one continuous-filament yarn have significantly increased the potential uses of rayon. Avron has high strength, high elongation, and high abrasion resistance and is particularly suitable for blending with other man-made fibres. Avril has a high wet modulus and is as inherently stable as cotton. Avlin has a flat fibre cross-section and permits a durable modification of handle without the use of surface resins when blended with either cellulosics or man-made fibres. Avicron is a latent-crimp continuous-filament yarn activated by wet processing. The dyeing and finishing characteristics of each of these rayons are discussed. S.B.D.

Dulling and Relustring of Cellulose Acetate

G. Rivat, P. Rochas, and S. Pierret

Bull. Inst. Text. France, (90) (Sept–Oct 1960) 25–37

Dulling of cellulose acetate by terpineol, isopropanol, and isobutanol is due to the formation of vacuoles which considerably increase the "empty space" (as defined by Hermans) in the fibre. Dulling also causes macromolecular rearrangements which may differ according to the reagent used— thus elongation at break is increased by terpineol, virtually unchanged by isopropanol, and decreased by isobutanol. Relustring with ethyl lactate and monochloroacetic acid produces new macromolecular rearrangements: the vacuoles disappear and the "empty space" in relustred fibres is similar to that in the original fibres. J.C.F.

Degree of Polymerisation and its Distribution in Cellulose Rayons. X— Tenacity and Breaking Extension of Monofilaments

R. J. E. Cumberbirch and C. Mack

J. Textile Inst., **51** (Nov 1960) T 458–T 483

A simplified model structure for cellulose regenerated from secondary cellulose acetate is described; theoretical relations of breaking strength and extension to degree of polymerisation (DP) are derived and agree well with experimental results. The fitting of these relations enables estimates to be made of crystallite size and of percentage crystallinity. It is also shown that a negligible fall in DP can be expected to occur on the mechanical rupture of a filament. W.G.C.

Effect of Nuclear Radiation on Fibrous Material
O. Teszler and H. A. Rutherford
Proc. U.N. Intern. Conf. Peaceful Uses At. Energy, 2nd, Geneva, 1958, **29**, 228–232

Yarns were subjected to neutron bombardment at thermal fluxes of approx. 2×10^8 and $1{\cdot}5 \times 10^{12}$ neutrons/cm²/s and γ-irradiation with dose rates of $4{\cdot}1 \times 10^4$ and $5{\cdot}0 \times 10^6$ r/h. The effects of the 4 radiation sources on the degree of polymerisation and other properties of the polymers were a function of total dose and independent of dose rate. Reactor and γ-sources were equally effective in modifying polymer properties at equal calculated dosages. Wool, silk, cellulose acetate, and cotton show no improvement in properties or are primarily degraded, and are decreasingly stable in the order given. Undrawn Orlon, undrawn nylon 6 and undrawn Dacron undergo one or more favourable changes. C.O.C.

Effect of High-energy Radiation on Wool Keratin and Polyamide and Polyester Fibres
H. Zahn, E. R. Fritze, H. Pfannmüller, and G. Satlow
Proc. U.N. Intern. Conf. Peaceful Uses At. Energy, 2nd, Geneva, 1958, **29**, 233–237

Neutron and γ-irradiation (doses of $0{\cdot}5 \times 10^{17}$ n/sq.cm and 10^7 r from a ^{60}Co source) was carried out in presence of air on wool, Diolen, Dralon, Perlon, wool–synthetic fibre blends, and wool modified with 1,5-difluoro-2,4-dinitrobenzene and benzoquinone. Changes in chemical analyses and physical properties were investigated. It seems that wool is more stable than nylon and less stable than polyester and acrylic fibres. When untreated wool is subjected to high-energy radiation, the tryptophan, tyrosine, and cystine components are especially vulnerable, leading to decreased cross-linkage and to formation of water-soluble degradation products. C.O.C.

Effect of Ionising Radiation on Fibre Keratin
D. Hildebrand and H. Würz
Melliand Textilber., **41** (Oct 1960) 1229–1231

Irradiation with neutrons causes degradation of cystine, tyrosine, and tryptophan, the last-named being most readily attacked. The extent of degradation is proportional to radiation intensity. Experiments with γ-rays were carried out in air and under vacuum. Tryptophan and tyrosine are attacked both in presence and in absence of oxygen, while cystine remains unaffected by irradiation with 8×10^6 rad in absence of oxygen. Low-intensity radiation (both neutrons and γ-rays) leaves the cystine content unchanged but increases the solubility in alkali, which indicates splitting of the main chains. W.M.

Bilateral Structure of Wool
M. Horio, T. Kondo, K. Sekimoto, and A. Teramoto
Z. Naturforsch., **15b** (1960) 343–345
Chem. Abs., **54** (10 Nov 1960) 23342

Histochemical determination of the isoelectric points of the two cortex components of wool dyed with acid and basic dyes gave values of 6·75 and 6·40 for the acidophilic part (I) on the inside of the curvatures ("A" cortex) for Merino and Corriedale wool, respectively. For the basophilic part (II) on the outside of the curvatures ("B" cortex) the values were 5·90 and 6·15. I and II differed in ultraviolet absorption, I absorbing more than II at 3130 Å, while II absorbed more at 2800 and 2650 Å. The hair root behaved like II toward basic dyes; I appeared at one-third of the distance between the root and the skin surface. C.O.C.

Microscopic, Microchemical, and Physical Examination of the External and Internal Structure of Wool. II— Experimental Investigation of the Bilateral Structure and its Relation to Fibre Variations and Damage
R. Ritter and H. Reumoth
Z. ges. Textil-Ind., **62** (1960) 342–350
Chem. Abs., **54** (10 Nov 1960) 23341

Diamine Sky Blue FF (C.I. Direct Blue 1) is especially suitable for differentiating the bilateral structure. Wool slubbing was dyed with 1% of the dye, dried, treated with 1% Na isopropoxide in isopropanol, the alcohol evaporated, and the fibre embedded in oil of bergamot. After 12–24 h, the ortho-cortex became colourless while the para-cortex remained blue. When the wool was dyed in a cold bath with Procion Brilliant Red 2BS (C.I. Reactive Red 1), and 1% NaOH added, the ortho-cortex was colourless to light yellow and the para-cortex yellow to orange, regardless of chemical or physical damage to the wool. Oxidation by H_2O_2 attacks both ortho- and para-cortex. The bilateral structure of wool is also rendered visible by treatment with a methanolic solution of LiBr, the scale structure being retained. LiBr seems to protect the fibre during aftertreatment with alkali. Apparently, appearance of the bilateral structure indicates that the fibre has previously been damaged. C.O.C.

Rates of Adsorption of Sulphuric Acid on Wool in Presence of Surface-active Agents
N. Buchsbaum and A. Datyner
J.S.D.C., **77** (Jan 1961) 16–22

Nitrosation of Tyrosyl Residues— Effect on some Properties of Wool
M. Leveau, M. Caillet, and N. Demonmerot
Bull. Inst. Text. France, (90) (Sept–Oct 1960) 7–24

Treatment of wool with nitrous acid, (*a*) in absence, and (*b*) in presence, of cupric ions reduces its solubility in urea–bisulphite. In contrast, (*a*) reduces the alkali-solubility of wool, whereas (*b*) increases it. It is concluded that nitrosation of tyrosyl residues in (*a*) leads to a reinforcement of tyrosyl interactions, whereas (*b*) introduces inter-chain linkages involving Cu which are stable to urea but unstable to alkali. Nitrosation of peptide groups may also have an insolubilising effect on wool. J.C.F.

Reaction of Lubricants with Wool
M. Kehren and H. G. Otten
Z. ges. Textil-Ind., **62** (1960) 314–319, 458–462, 477–480
Chem. Abs., **54** (10 Nov 1960) 23341

Work which confirms that wool is not damaged by the combined action of the lubricant and photo-oxidation. C.O.C.

Ultrasonic Degradation of Wool and Hair
J. H. Bradbury *Nature*, **188** (15 Oct 1960) 207–208

Mechanical degradation of wool and hair by ultrasonic methods at 16–24 and 40 kc/s has been observed. Microscopical examination of residual fibres showed scale damage extending from zero to complete removal of scale edges. Merino 90s was not apparently damaged, Lincoln 36s showed some damage, but not as great as human hair. Yields of degraded wool were 0·6% in neutral and alkaline conditions and less in acid conditions, this material consisting of 90% of flat cuticle pieces and 10% needle-like cells of sizes up to cortical cells. Chemically modified Merino 36s top, e.g. by acid chlorination and other methods, showed no difference in ultrasonic breakdown yields when compared with untreated top. There was no detectable change in amino-acid content after ultrasonic treatment. F.J.

Making Wool a More Useful Fibre
B. C. M. Dorset *Text. Manuf.*, **86** (Oct 1960) 407–411

A review of recent developments, including resin deposition, newer scouring treatments, temperature effects, resistance to pilling, and difficulties encountered in the production of fast-dyed mixtures with acrylic fibres. M.T.

Man-made Fibres— Some Recent Developments
A. J. Hall *Fibres & Plastics*, **21** (Dec 1960) 355–357, 368

The modification of polyamide and polyester fibres is considered, with particular reference to the use of derivatives of durene (1,2,4,5-tetramethylbenzene) and piperazine. Recently developed techniques to overcome the problems of static electricity and pilling are also described. J.W.D.

Dyeability of Polyacrylonitrile
G. F. D'Alelio and L. X. Mallavarapu
Makromol. Chem., **38** (1960) 56–71 (*in English*)
Chem. Abs., **54** (10 Nov 1960) 23413

Review, 219 references. C.O.C.

Manufacture and Dyeing of Polyamide Fibres— II
R. S. Asquith and G. E. Styan
Fibres & Plastics, **21** (Dec 1960) 365–368

The preparation of a number of different polymers, including nylons 6, 6.6, 6.10, and 11, and their chemical and physical properties are dealt with. Improved methods of spinning are also referred to. There are two photographs and several references. J.W.D.

Coating Fabrics with P.V.C.

E. Mattinson *J. Textile Inst.*, **51** (Dec 1960) P 690–P 698

The production of certain industrial fabrics, e.g. tarpaulins, tentage and ventilation ducting, by coating woven fabrics with polyvinyl chloride is described. The design of suitable cloth structures, the effect of coating on the tensile and tear strengths and the methods of bonding P.V.C. to man-made fibres are also discussed. W.G.C.

PATENTS

Stabilising Saponified Stretched Cellulose Ester Fibres to Heat

Celanese Corpn. of America

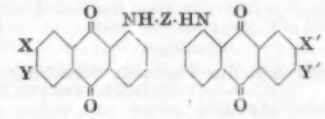
BP 854,920 (U.S.A. 5 Apr 1956)

Incorporation of a Ca, Ba, Sr or Ca carboxylate, e.g. Ca lactate or Sr caproate, improves the resistance to heat of saponified stretched cellulose ester fibres. C.O.C.

Mass Coloration of Cellulose Acetate and Synthetic Polymers

S *BP* 859,339 (Switzerland 27 June 1957)

Cellulose acetate is mass-coloured yellow of good light fastness by dyes of formula—



(X, X′, Y, Y′ = same or different H, F, Cl, Br; Z = dicyl radical of benzene-1,4-dicarboxylic acid, 1,1′-azobenzene- or 1,1′-azoxybenzene-4,4′-dicarboxylic acid, or of an ethylene-1,2-dicarboxylic acid, optionally substituted by CH_3 or Cl). Thus, a mixture of sec. cellulose acetate (100), acetone (400) and dye (1) (X = X′ = Y = Y′ = H; Z = radical of 1,1′-azobenzene-4,4′-dicarboxylic acid) is ground in a ball-mill until particle size is $< 1\ \mu$. The spun filament is yellow of outstanding light fastness.

BP 859,343 (Switzerland 16 Aug 1957)

Compounds of the above formula are used for mass-coloration of synthetic polymers, e.g. polyvinyl chloride, polystyrene, polyesters, and also in the melt spinning of polyamides and polyesters. They have excellent fastness to light, migration, water, washing, cross-dyeing, chlorine in acid and alkaline medium, chlorite, sublimation, oxalic acid, dry cleaning, gas-fumes, rubbing, pressing, peroxide bleaching, blind vatting, perspiration, heat, and hydrosulphite. Thus, a mixture of the dye (1) (X = X′ = H, Y = Y′ = Cl; Z = radical of 1,1′-azobenzene-4,4′-dicarboxylic acid), the copolymer of 60·1% vinyl chloride and 39·9% acrylonitrile (199) and acetone (800) is mixed at 50°C for 4 h and the solution extruded into water to give a yellow filament of excellent light fastness. A.T.P.

Red Mass-coloured Cellulose Acetates

Sz *BP* 857,130 (Switzerland 24 Dec 1957)

Cellulose acetates (I) are mass coloured using dyes (II)—

(A may contain Cl or CH_3), obtained by condensing perylene-3,4,9,10-tetracarboxylic acid or anhydride with the requisite amine (e.g. *p*-chloraniline, *p*-toluidine). II must be finely-ground and preferably a mixture of II, I and a solvent for I (but not for II) is ground to $< 1\mu$ before extrusion, I is coloured red of excellent fastness to light, washing, acid cross-dyeing, chlorine in acid medium, dry cleaning, and peroxide bleaching. A.T.P.

Cellulose Anthranilate Fibres

U.S. Secretary of Agriculture

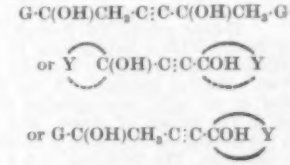
USP 2,926,063 (24 July 1958)

Cellulosic fibres containing hydroxyl groups are treated with a water-miscible organic liquid, e.g. dimethyl sulphoxide, dimethyl formamide, acetic acid, and acetone, containing by wt. 0·1–12·0% of a strong base and 1·0–3·0% of isatoic anhydride. The treated fibres are then heated at 50–170°C for 2–30 min until esterification occurs and the fibres contain 1 anthranilate group per 2–30 anhydroglucose units. Thus cotton sheeting was padded with its own weight of a solution containing NaOH (0·37), water (11), isatoic anhydride (14·7), and dimethylsulphoxide (74). It was divided into three parts, A, B, and C. A was heated in a forced draught oven for 30 min at 50°C, B for 3 min at 130°C, and C for 6 min at 130°C. All were then washed in running hot water for 2 h, dried and finally conditioned for 16 h. B and C gained 9% in weight and A slightly less. Their handle was similar to that of the untreated cloth. They all dyed readily with Kiton Fast Red 3GLL. The treated cloth has good resistance to attack by microorganisms and may be diazotised and coupled to yield highly coloured fabrics. C.O.C.

Treating Acrylic Fibres with Acetylenic Glycols

Dow Chemical Co. *BP* 852,402 (U.S.A. 16 Apr 1958)

In the wet-spinning of acrylic fibres, application as opening agents to the aquagel fibres of compounds of the following formula—



(G = Alk of 1–4 C; Y = cyclising linear polymethylene unit which with the C to which it is attached forms a 5- or 6-membered alicyclic ring), e.g. 4,7-dimethyl-5-decyne-4,7-diol, and then drying in hot air at 100–150°C for 5–30 min, prevents sticking together of the yarns during drying. The opening agent is entirely removed during the drying, probably by steam distillation, and so has no effect on subsequent processes. C.O.C.

Stabilising Polyamides

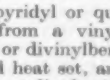
Phrix-Werke *BP* 858,843 (Germany 23 Dec 1957)

Incorporation of a mixture of an aminoalcohol or its amide ester and an ester, amide or salt of an oxyacid of phosphorus, e.g. a mixture of ethanolamine and Na phosphate, stabilises polyamides to ageing under the influence of heat, air, moisture, and light. C.O.C.

Polyvinyl Alcohol Fibres of Improved Dyeability

Kurashiki Rayon *BP* 855,552 (3 June 1957)

Oriented fibres of good dyeability are obtained by using as the raw material polyvinyl alcohol (65·0–99·5% by wt.) and a polymerised basic vinyl compound of which $\leq 20\%$ by wt. consists of units of—



(Py = pyridyl, alkylpyridyl or quinolyl), the remainder being units derived from a vinyl ester, acrylonitrile, vinyl chloride, styrene or divinylbenzene. After extrusion they are stretched and heat set, and then 5–50% of the hydroxyl groups are acetalised with a monoaldehyde of < 21 C. C.O.C.

Stabilising Vinyl Lactam–Acrylonitrile Copolymer Fibres to Light

Dow Chemical Co. *USP* 2,922,693 (28 Dec 1956)

Treatment with an aqueous solution containing 8% hydroxylamine sulphate and 0·5–5·0% H_2SO_4 on the weight of the fibre yields fibres which do not discolour on long exposure to ultraviolet radiation. C.O.C.

Pigments from Perylene-3,4,9,10-Tetracarboxylic Anhydride (IV p. 128)
Pigmented Coatings for Glass Fibre (V p. 131)
Irradiation of Synthetic Linear Polymerisation or Polycondensation Products (X p. 142)
Use of Resins in Bonded Fabrics (XIII p. 144)
Stabilised Acrylonitrile Polymers (XIII p. 144)
N-Vinyl-X-alkyl-2-oxazolidines and their Polymers (XIII p. 144)
Vinyl and Vinylidene Polymers Pigmented with Carbon Black (C.I. Pigment Black 6 and 7) (XIII p. 144)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Electron-microscopic Study of the Degumming of Silk

M. Minagawa *J. Soc. Textile Cellulose Ind. Japan*, **16** (June 1960) 507–513

Silk can be readily enzymatically degummed at pH 6–8 and 40–45°C in 1 h, compared with the 2–3 days required for fermentation degumming. The enzyme does not cause discoloration and the appearance of the fibre surface under the electron microscope and determination of the section removed revealed more complete and superior degumming than that obtained by use of papain or trypsin. Bacterial enzyme degumming is shown in electron photomicrographs to differ from that obtained with Marseilles soap, Na_2CO_3, or ammonia. The enzyme method can be safely applied to all types of silk as it selectively decomposes the sericin leaving the fibroin unattacked. It results in very even degumming. C.O.C.

Continuous Bleaching of Fabrics in Open Width

M. G. Kachurin, L. A. Gotovtaeva, and M. G. Shikher *Tekstil. prom.*, No. 9 (Sept 1960) 40–44

The authors describe their experiences in continuous bleaching of cellulosic fabrics using a Benteler installation composed of two identical sections. In the first section fabric is heated in an ager, impregnated with caustic alkali, exposed to the action of steam, and thoroughly washed off. In the second section fabric is similarly treated with hydrogen peroxide. The total time is 3–4 h and at all stages the fabric is kept in open width. Steaming is the most critical operation and, if non-uniform, can introduce dyeing variations and affect the strength of the fabrics. Two methods of bleaching were used— (*a*) fabrics for white and pastel shades were given both alkaline and peroxide treatments, (*b*) fabrics for medium and heavy shades were given an alkaline treatment only. The use of the second section of the installation for continuous dyeing is suggested. L.S.L.

Corrosion Problems in Chlorite Bleaching

K. Günther *Melliand Textilber.*, **41** (Oct 1960) 1237–1243

In spite of the advantages of sodium chlorite as a bleaching agent, its use has so far been limited by the difficulty of finding materials from which bleaching plant can be made that are sufficiently resistant to corrosion. Titanium is too expensive and too difficult to work. Efforts to use stainless steel of different types, together with corrosion inhibitors (nitrate, fluorine compounds, etc.) or to prevent corrosion by electric polarisation were only partly successful. Replacing the acid used as activator by slowly hydrolysing organic esters gives less good bleaching effects without eliminating corrosion. To overcome the corrosion problem an open-width bleaching apparatus suitable for continuous operation has been built entirely of ceramic material. After being padded with the bleaching liquor in an impregnating vessel connected to a storage tank by pump, the fabrics pass through a squeezing device into a vertical steam chest and from there into a cylindrical chamber where they are rolled up. The chambers can be heated and are detachable from the bleaching machine. Each chamber has its own motor so that the batched-up fabrics can be kept rotating for any desired time. Bleaching recipes are given. W.M.

Hydrogen Peroxide Bleaching of Cotton

L. Chesner (reply A. S. Ramadan) *J. Textile Inst.*, **51** (Nov 1960) T 487–T 488

In his article in the June issue of *J. Textile Inst.* (J.S.D.C., **76** (Sept 1960) 568) Ramadan disagrees with previous work by Chesner on the effect of grey cotton cloth on the stability of hydrogen peroxide bleaching solution. Chesner now points out that the bleaching conditions which he used (J.S.D.C., **74** (1958) 531) were different from those used by Ramadan and referred to laboratory and not works-scale bleaching. Ramadan, in reply, agrees with this conclusion and quotes earlier work (J.S.D.C., **75** (1959) 385) in support of his view that the stabilising effect of grey cotton impurities is operative only in a bath which is otherwise unstable. W.G.C.

Preparation and Use of Peracetic Acid in Bleaching

G. Rösch *Deutsch. Textiltech.*, **10** (1960) 191–195 *Chem. Abs.*, **54** (25 Sept 1960) 18964

The dilute peracetic acid required for bleaching can be prepared *in situ* by reaction between acetic anhydride and H_2O_2. Best yield of peracetic acid (80%) occurred after 4 h reaction between H_2O_2 (1 part) and acetic anhydride (6). $Na_4P_2O_7$ (1–3 g/l.) was the most efficient stabiliser, followed in order of decreasing efficiency by 1 g/l. Calgon T, 1 ml waterglass and 0·1 g/l. $MgSO_4$, 1 g/l. $CaCl_2$, and 1 g/l. Trilon B. After 1 h at pH 5 or 6, enough acid remained to bleach, and bleaching was satisfactorily completed in 2 h; at pH 8 no acid remained after 1 h. Solutions of 3·12–3·19 g/l. peracetic acid stabilised with 1 g/l. $Na_4P_2O_7$ bleached best at 80°C. Cotton was bleached in a bath containing 100% peracetic acid (3 g/l.), Calgon T (0·5), $Na_4P_2O_7$ (1) and Concentrate W50 (1), brought to pH 6 with NaOH, raised to 80°C in 1 h and kept for 1 h at that temperature. The pad–steam method yielded poorer whites, but freedom from impurities. Peracetic acid has the same stripping effect on many dyes as has $NaClO_2$ and it removes about 80% of any size present. Peracetic acid gave appreciably improved bleaching of Dederon, but is of no use with polyester fibres or Wolcrylon. Cotton was successfully bleached at room temperature with 1·5–2·0% peracetic acid, but it required $\leqslant$ 4 h at pH 5–6 under which conditions the acid decomposes rapidly. For cold bleaching it is better to place the NaOH in the bath first and then add the peracetic acid. C.O.C.

PATENTS

Stabilisers for Alkaline Peroxide Bleaching Baths

DuP *USP* 2,927,082 (19 Jan 1956)

Water-soluble gluconate added to alkaline peroxide baths chelates any Mg ions present and prevents or delays precipitation of insoluble magnesium compounds. The resulting Mg-gluconate chelate is an effective stabiliser for the solution over a wide pH range and is particularly useful at pH 11–14. C.O.C.

Bleaching Hair

L. R. Bruce *BP* 852,777 (9 Apr 1958)

When hair is bleached with a paste containing H_2O_2, presence of a phthalocyanine complex of a metal of atomic number 27–30, e.g. Cu phthalocyanine, results in more uniform bleaching, less damage to the hair, and prevention of yellow or reddish tinges. C.O.C.

VIII— DYEING

Development of the First Modern Continuous-dyeing Process and its Relation to other such Methods

J. Müller *Melliand Textilber.*, **41** (Oct 1960) 1253–1259

The first continuous process for dyeing with vat dyes was developed in a dyeworks near Vienna in 1938–1944. Vat acids were used, since very fine dispersion was essential and powder brands sufficiently fine were not available. The process consists of the following steps: padding fabrics with vat acid dispersion with addition of thickeners to counteract migration; drying; padding with hydrosulphite + NaOH. Fabrics leave the second padder with 140% expression and enter the Elektrofixierer, where they are quickly heated. Steam is produced from the water evaporated from the fabrics. After passing through an air space, the fabrics run into an open-width developing and soaping range. Very level dyeings, good penetration, and consistent shades throughout very long runs were obtained. The other continuous processes for dyeing with vat dyes— excepting the Standfast Molten Metal Process— are considered to be modifications of the Vat Acid–Elektrofixierer method. W.M.

Dyeing Properties of Azo Acid Dyes. VI— Conductivity of Free Dye Acid Solutions

T. Iijima and M. Sekido *J. Soc. Textile Cellulose Ind. Japan*, **16** (July 1960) 571–574

The conductivity of the free dye acid solutions at 25, 50 and 90°C was measured of the following dyes: (*A*) naphthionic acid→β-naphthol, (*B*) α-naphthylamine-G acid and (*C*) naphthionic acid→G acid. The equivalent conductivity was plotted against the square root of the concentration. With all three dyes the curvature of the graph has a maximum at 25°C, indicating that all three dyes are present as ionic micelles. With increase in temperature the McBain effect tends to disappear and at 90°C all three dyes give a straight line showing that they are all in true solution. These dyes have the same skeletal structure differing only in the number of SO_3H groups

(*A*—one, *B*—two, *C*—three). The fewer the SO_3H groups the greater the tendency to form ionic micelles in aqueous solution. C.O.C.

Azoic Combinations on Cotton
P. J. Derwent
Textile J. of Australia, **35** (20 Aug 1960) 654–658

Azoic coupling components, bases and dyeing salts, and their application to cotton are discussed at length. Precautions necessary in each stage of the process when it is carried out using different types of machinery, e.g. jig, winch, package dyeing and padder, are detailed. W.P.M.

Direct Dyeing of Cellulose
K. Nishida
VIII— Relation between Dyeing Properties and Conjugated System of Double Bonds (II)
J. Soc. Textile Cellulose Ind. Japan, **16** (June 1960) 484–485

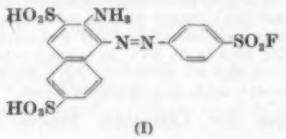
The absorption spectra of Congo Red (C.I. Direct Red 28) are given in solution and when dyed on regenerated cellulose film. The peak of the absorption band in the spectrum of the dyeing is at a longer wavelength (7·5 mμ in 3800 Å) than it is in the spectrum of the solution. The energy change on displacement of the absorption band of the triazole derivative of Congo Red in regenerated cellulose film is the same as that for common direct dyes.

IX— Relation between Dyeing Properties and Conjugated System of Double Bonds (III)
Ibid., (July 1960) 575–578

The absorption isotherms of Zambesi Black D (C.I. 27700) and aniline→*p*-cresidine→γ-acid determined under identical conditions showed the two dyes to have nearly the same mean standard affinity (4·4 kcal). Zambesi Black D contains a longer conjugated chain of double bonds than does the other dye. The results are explained by taking into account the hydration of the dye. C.O.C.

Nature of the Bond between a Reactive Dye and Cellulose
B. Krazer and H. Zollinger
Helv. chim. Acta, **43** (Oct 1960) 1513–1522

Evidence for the covalent character of the bond formed between reactive dyes containing a sulphonyl fluoride group and cellulose is presented. The evidence is based on the application of a reaction used in carbohydrate chemistry for the determination of primary hydroxy groups, in which all hydroxy groups are esterified by the action of *p*-toluenesulphonyl chloride, the tosyl ester so formed then being treated with sodium iodide in acetone, when esterified primary hydroxy groups are replaced by iodine atoms, esterified secondary groups remaining unchanged. Cotton was dyed with the reactive dye (I), this being regarded as ester formation as with *p*-toluenesulphonyl chloride, and then was subjected to the action

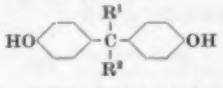


(I)

of sodium iodide in acetone. Replacement of esterified hydroxy groups by iodine took place, and was found to be specific for the sulphonyl fluoride dyes. From the extent of replacement it was concluded that ca. 75% of the dye reacted with primary hydroxy groups and the remainder presumably with the secondary hydroxy groups in the 2- and/or 3-positions in the glucose units. I.G.L.

Affinity of 4,4′-Dihydroxydiphenyl Alkanes for Cotton
V. N. Klyuev and T. S. Meshkova
Tekhnol. tekstil. prom., No. 3 (16) (1960) 113–117

The use of 4,4′-dihydroxydiphenyl alkanes of formula—



where $R^1 = R^2 = H$ or CH_3; $R^1 = H$, $R^2 = CH_3$; $R^1 = CH_3$, $R^2 = C_2H_5$; $R^1 + R^2 = (CH_2)_5$, as azo components has been investigated. Affinities determined by standard techniques are very close to those of common azo components, e.g. deriv. of β-hydroxynaphthoic acid. G.J.K.

Adsorption of Non-ionic Dyes by Cellulose
J. R. Aspland and C. L. Bird J.S.D.C., **77** (Jan 1961) 9–12

Dyeing Cellulose Acetate with Disperse Dyes. X— Saturation Values with Mixtures of Dyes
C. L. Bird and P. Rhyner J.S.D.C., **77** (Jan 1961) 12–16

Dyeing Fibres containing Active Methylene Groups IV— Partial Acetoacetylation of Viscose Rayon
H. Iida and K. Konishi
Rept. Govt. Chem. Ind. Research Inst. Tokyo, **55** (Sept 1960) 351–355

Viscose rayon cloth was soaked in a soln. of 2 mol/l. sodium acetate, dried for 24 h at room temp., and allowed to react with 10% diketen soln. at 60°C. Partially acetoacetylated cloths containing 2–5% combined diketen by weight were obtained in as little as 2 min.

V— Dyeing Partially Acetoacetylated Viscose Rayon
H. Iida and N. Kuwabara *Ibid.*, 356–359

Cloths prepared by method of part IV, previously dyed with a diazotisable direct dye, were immersed in sodium nitrite soln. acidified with acetic acid. The wash fastness of the dyeings was thereby improved. It is concluded that the diazotised direct dye is bonded to the acetoacetyl group of the partially acetoacetylated viscose rayon. The dyes used were C.I. Direct Orange 84, C.I. Direct Red 153, C.I. Direct Green 43 and 48, C.I. Direct Blue 140, and C.I. Direct Black 86. Most metal complexes of formazan dyes formed by reaction of *o*-aminophenol derivatives with the cloths gave dyeings fast to light and to washing. M.T.

Introduction of Chelate Bonds into Fibres. XIII— Energy of Activation in Adsorption of Dyes by Mercury-treated Wool
N. Hōjō *J. Soc. Textile Cellulose Ind. Japan*, **16** (June 1960) 479–483

The dyeing of mercurised wool with Acid Orange II and Chrome Orange A proceeds in three steps: (*a*) absorption of dye on the fibre surface, (*b*) diffusion of absorbed dye into the fibre, (*c*) chemical combination between the dye and the mercury ions in the fibre. Diffusion of the dye into the fibre determines the rate of dyeing. Determination of the energy of activation of dyeing indicates that the application of these dyes to mercurised wool requires more energy than application to untreated wool. Probably, cross-links formed during the chelation of the dye with the mercury in the wool, and the change of charge on the fibre, have some bearing on these observations. C.O.C.

Batch-wise Dyeings of Silk with Dichlorotriazine (reactive) Dyes
A. D. Virnik and H. A. Chekalin
Tekstil. prom., No. 9 (Sept 1960) 45–46

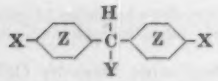
Four methods for dyeing silk with reactive dyes suggested in I.C.I. Technical Information Leaflet Dyehouse No. 348 have been examined in detail. Quantities of dye chemically attached to the fibre have been estimated after thorough washing of dyed samples with aqueous pyridine, drying, dissolving in cold hydrochloric acid, and examination of coloured solution colorimetrically. L.S.L.

Dyeing Suède Pigskins
F. Stather, H. Herfeld, H. Moser, and K. Härtewig
Ges. Abhandl. deut. Lederinst, Freiberg/Sa., (14) (1959) 61–76
Chem. Abs., **54** (10 Oct 1960) 20265

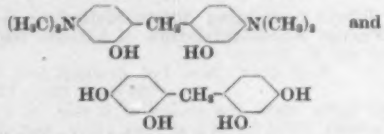
The chrome retanning of the previously dried leather which is necessary to impart sufficient affinity for anionic dyes can be replaced by treatment with a cationic resin. Topping with a basic dye, dyeing at > 70°C and exhausting the dyebath by adding a cationic auxiliary agent are all disadvantageous. The suitability of 44 different dyes is listed. C.O.C.

Dyeing of Difficultly Dyeable Fibres
K. Yamada
XII— Diphenylmethane Condensation Dyes
J. Soc. Textile Cellulose Ind. Japan, **16** (June 1960) 486–492

A study of the dyeing of vinylon with 12 diphenylmethane compounds of formula—



together with an aromatic amine, phenol, or aminophenol, and using a quinone as oxidising agent. Many diphenylmethane compounds react with amines or phenols and chloranil to form triphenylmethane dyes, but



which have hydroxy radicals in 2,2′-positions, do not react with amines or phenols, but form xanthene dyes with chloranil or other oxidant. The reactivity of the substituents in the compounds of the above formula is X = $N(CH_3)_2 > N(C_2H_5)_2 > NH_2$; Y = OH > NH_2 > H; Z = CH_3 > H > NH_2 > NO_2. With the amines and phenols the order of reactivity is (1) 1,3,5-benzenetriol > 1,3-benzenediol > phenol > 1,4-benzenediol; (2) *NN*-dimethylaniline, *NN*-diethylaniline, *N*-ethylaniline, *N*-phenylaniline > aniline; (3) *NN*-dimethyl-*m*-hydroxyaniline > *m*-hydroxyaniline > aniline > *m*-nitroaniline; (4) 1-naphthol > 2-naphthol. 4,4′-Bisdimethylaminobenzhydrol and 4,4′-bisdimethylaminobenzhydrylamine are best applied by a three-bath method, i.e. each component is applied separately, but 4,4′-bisdimethylaminodiphenylmethane is best dyed with all the components in the same bath. In all three cases, chloranil is a better condensing agent than *p*-benzoquinone.

XIII— Fixing Basic and Condensation Dyes with Phosphomolybdic or Phosphotungstic Acid

Ibid., 492–495

Vinylon can be dyed by treating it with phosphomolybdic, phosphotungstic, or phosphomolybdotungstic acid and then dyeing with a basic or condensation dye. The complex acid is usually made up of 1:3 P_2O_5:Mo- (or W)O_3, and in the case of phosphotungstic acid the pH should be < 2. Very brilliant dyeings of good fastness to light and to washing are obtained. The phosphotungstic dyeings have better light fastness than the others. The phosphomolybdic dyeings quickly darken or turn green but after that are less sensitive. The phosphomolybdotungstic dyeings are intermediate in fastness between the other two. C.O.C.

XVI— Dyes formed by Condensation of Thioindoxyl Carboxylic Acids and Quinones

Ibid., **16** (Sept 1960) 753–758

Dyes formed by condensation of thioindoxyl carboxylic acids with β-naphthoquinone, sodium β-naphthoquinone 4-sulphonate, acenaphthenequinone, *p*-benzoquinone, chloranil, and α-naphthoquinone were applied to Saran, Terylene, and Farlon. The dyes formed from the first three quinones gave dyeings with high chroma, but because of their low light fastness they are of no practical interest.

XVII— Dyes formed by Condensation of Thioindoxyl Carboxylic Acids and Isatins

Ibid., 758–762

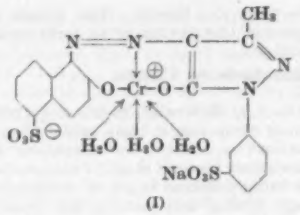
Thioindigo Scarlet R dyes are formed and used to dye synthetic fibres. The effect of substituents in the isatin molecule is small, but the homo-compounds yield dyes of higher affinity for Farlon fibre giving dyeings of improved light-fastness, whereas the nitro derivative gives a dyeing of lower fastness on this fibre. The light-fastness is lowest when thioisatin is used. The dyes are particularly suitable for Saran and Farlon. M.T.

Dyeing of Synthetic Fibres. X— Investigation of Reactions between Kapron and Premetallised Dyes

A. A. Kharkharov and E. E. Starikovich

Tekhnol. tekstil. prom., No. 4 (17) (1960) 117–124

Two premetallised dyes, Acid Pink M (1:1 complex) (I) and Lightfast Acid Orange NZKM (1:2 complex) (II) were applied to normal, hydrolysed, and deaminated Kapron, the pH and dye concentration being varied; dyeings were taken to equilibrium. In the case of 1:1 complexes, the dye is said to be attached to the fibre by salt



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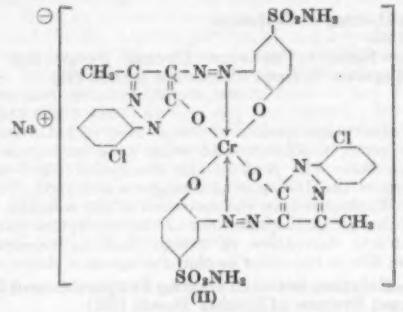


(II)

linkages and to a lesser extent by co-ordination linkages. Reaction with 1:2 complexes depends only partly on the presence of amine end-groups. The main link between dye and fibre is through hydrogen bonds and van der Waals forces. L.S.L.

Dyeing Acrylic Fibres in Presence of Univalent Copper

A. A. Geller and A. B. Pakshver

Khim. Volokna, (3) (1960) 19–21

Chem. Abs., **54** (10 Nov 1960) 23337

Cu^+ and Cu^{2+} react with the acid groups of acrylic fibres to form salt-like compounds in the proportion of about 6×10^{-5} g-equiv/g of fibre. An acid dye is bound by a secondary valence in case of Cu^+ and by a primary valence in the case of Cu^{2+}, when the fibre is dyed in two baths with intermediate drying. Sorption of the dye is considerably reduced in the second bath. Salts of Cu^{2+} give dull colours in dyeing because of deposition of Cu compounds of the dye. C.O.C.

PATENTS

Azoic Dyeing

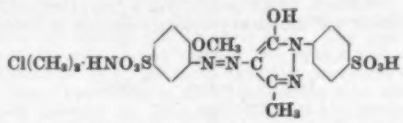
Whiffen & Sons *BP* 854,341 (24 Jan 1958)

Fabric is impregnated with a diazotised amine, treated with urea and then with a coupling component. Thus nylon dyed with Dispersol Diazo Black 2BS (C.I. Disperse Black 2) is diazotised, rinsed first in cold water, and then in 0·2% aqueous urea brought to pH 5–6 with HCl. It is then treated for 30 min at 30°C in a bath containing 1% phloroglucinol on the wt. of nylon, 2 g/l. Na acetate, and 2 ml/l. 30% acetic acid, and finally soaped. C.O.C.

Reactive Dyes for Cellulosic Fibres and their Application

Ciba *BP* 852,960 (Switzerland 14 Aug 1956)

Cellulosic fibres are fast dyed by applying dyes (stilbene, azine, dioxazine, anthraquinone, and especially acid phthalocyanine and metal-free and metalliferous acid azo) containing ≮ 1 acidic water-solubilising group and ≮ 1 substituent bound to the C atom of an aliphatic chain of a sulphonic acid alkylamide residue, and which is split off on alkali treatment, and finally fixing the dye by heat in presence of an acid-binding agent. Thus cotton is dyed yellow, fast to washing, by applying—



from an aqueous salt bath at 20–25°C and finally adding NaOH to the dye liquor and heating at 85–90°C.

A.T.P.

Dyeing Hair, Fur and other Keratinous Fibres with Oxidation Bases

Société Monsavon l'Oreal

BP 857,070 (France 15 Apr 1958)

The fibres are impregnated with an aqueous solution of 2,4-, 2,5-, or 3,4-dihydroxyaminobenzene or an acid addition salt thereof and then oxidised by air or other oxidising agent. When used as hair dyes these oxidation bases, individually or in mixtures, yield dyeings from blond to light brown and black. C.O.C.

Improving the Fastness to Light of Dyeings on Acrylic Fibres containing Vinyl Lactam Polymer

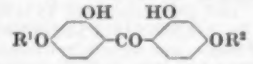
Dow Chemical Co. *USP* 2,922,692 (28 Dec 1956)

Dyeings made with direct, acid, and chrome dyes are given improved fastness to light by treating them with an aqueous solution containing cuprous ions at $> 70°C$. Treatment preferably follows immediately after dyeing, and the cuprous ions are best obtained by reducing cupric ions with hydroxylamine sulphate or an equivalent reducing agent. C.O.C.

Stabilising Dyed Polyester Fibres against Ultraviolet Radiation

General Aniline *BP* 852,977 (U.S.A. 26 May 1958)

Presence in the dyebath of a compound of formula—



e.g. 2,2′-dihydroxy-4,4′-dimethoxybenzophenone, gives good protection to the dyed fibre, as regards both colour and strength, against ultraviolet degradation. C.O.C.

Dyeing Textile Articles to give them Areas of Gradually Varying Colours

F. R. Frenzel *BP* 852,560 (Sweden 25 May 1957)

The article, e.g. a stocking, is drawn into a support frame having laterally spaced elongated members. A coating member parallel to the elongated members is forced up between the latter from outside to give the article an inverted U- or V-shape. Only the edges of the U- or V-shape are immersed in the dyebath and the article is lifted out of the bath while still in the U- or V-shape. C.O.C.

Open-width Dyeing and Finishing of Fabrics of Cellulosic, Man-made, or Blended Fibres (I p. 122)

Microscopical Staining of Fibre Cross-sections with Procion Black HGS (C.I. Reactive Black 1) (VI p. 133)

Affinity for Cellulose of Polyenedicarboxylic Acids of the Crocetin Type and of Quaternary Ammonium Compounds (VI p. 133)

New Cellulosic Fibres (VI p. 133)

Dyeability of Polyacrylonitrile (VI p. 134)

Manufacture and Dyeing of Polyamide Fibres— II (VI p. 134)

Printing and Dyeing Polyester Fibres with Disperse or Vat Dyes (IX this page)

Chromatographic Study of Some Procion Dyes (XIV p. 145)

Economical Dyehouse Management (XV p. 148)

IX— PRINTING

Automatic Screen Printing

F. Perutz

Amer. Dyestuff Rep., **49** (28 Nov 1960) 884–886

Of an estimated 458 automatic screen-printing machines in existence, only 30 are in the U.S.A., compared with 136 in Italy and France, and 277 in Europe as a whole. Automatic screen printing is compared with high-quality roller printing, but not with mass-produced prints, for labour costs, colour costs, production, and flexibility of operation. The specific features of several automatic screen-printing machines are discussed. S.B.D.

Use of Remazol Dyes in Printing

H. Dethloff *Tinctoria*, **57** (Nov 1960) 460–464

Different methods of application, and suitable thickeners and catalysts for the reaction between dye and fibre, are discussed. Remazol Salt FD, the practically neutral solutions of which split off alkali during steaming, is recommended instead of free alkalis, since it yields printing pastes of superior stability. As a new development, a two-phase printing process (printing, drying, padding) is described. Steaming for only 15–20 s is required; other advantages are claimed. Finally, the application of a new type of dye, Inthion Brilliant Blue 5GL, in water-soluble form is described. The solubilising group splits off during steaming and the dye is fixed in the fibre as an insoluble pigment. W.M.

Application of Ammoniacal Carboxymethylcellulose in Pigment Printing

A. S. Stepanov and A. G. Barinova

Tekstil. prom., No. 9 (Sept 1960) 47–49

Addition of ammoniacal carboxymethylcellulose to the printing paste was found to improve the fastness properties of the print. Carboxymethylcellulose is said to react with melamine–formaldehyde resin in the printing paste and to assist fixation of the pigment. The following recipe is suggested (per kg of paste): pigment paste 80 g, melamine–formaldehyde resin 100 g, 3% solution of ammoniacal carboxymethylcellulose 540 g, emulsion thickener 340 g. After printing the fabric is dried only. The drying apparatus has to be equipped with an efficient extraction system for removal of inflammable and toxic solvents. L.S.L.

PATENTS

Light-sensitive Coating for Preparing Printing Screens

W. A. Leeds and N. J. Sweete

USP 2,924,520 (25 June 1956)

The coating composition consists of a gelatinous material, e.g. hide glue (85 pt. by wt.) and $K_2Cr_2O_7$. The gelatinous material must have a bloom gel strength of > 400. Preferably 1–2 parts of the dichromate are replaced by the same amount of D&C Green No. 5 (C.I. Acid Green 25). This dye helps to provide the correct pH and facilitates maximum penetration of the coating by ultraviolet radiation. The coated screens may be prepared and stored for 6 weeks without loss of sensitivity to light. C.O.C.

Printing and Dyeing Polyester Fibres with Disperse or Vat Dyes

CFM *BP* 854,340 (Germany 17 Jan 1957)

Addition of sulphonated castor or sulphonated olive oil to the printing paste or padding liquor and then treatment of the printed or padded goods at 190–220°C for 30–120 s results in very deep prints or dyeings provided there is < 80 g of a 50% solution of the sulphonated oil present in each kg of printing paste or litre of padding solution. C.O.C.

Transparent Prints on Textiles by aid of Carbamic Resins

BASF *BP* 854,582 (Germany 12 Apr 1958)

Use of an oil-in-water emulsion containing 25–45% of a water-insoluble etherified hardenable carbamic resin dissolved in an organic solvent, and/or hydrocarbon, yields prints having a soft and non-sticky handle and prevents the printing rollers or stencils being gummed up relatively quickly. Thus aliphatic hydrocarbons of boiling pt. 140–220°C (230 parts) are emulsified in a mixture of water (10), 6% tragacanth solution (40), and 20% aqueous solution (120) of the reaction product of 1 mol. sperm oil alcohol and 25 mol. ethylene oxide. Into the resultant emulsion is stirred a 70% solution (500) in butanol of a water-insoluble hardenable etherified urea–formaldehyde condensate. This yields a stable viscous emulsion which when diluted with water, printed into fabric and then treated for 3 min with dry air at 120°C yields sharp transparent prints and a soft and non-sticky fabric. C.O.C.

Wrinkle Finish Printing

Commonwealth Engineering Co. of Ohio

BP 852,926 (30 Sept 1958)

One face of a sheet, e.g. cloth, paper or vinyl film, is treated with a wrinkle coating composition. The coated surface is then contacted with a metallic design, and the area of the sheet around the metallic design and the metallic design are heated. This produces a wrinkle design finish which differs from that of the wrinkle finish and/or colour of the surrounding area. The metallic design may be a printing plate or drum. C.O.C.

One-component Diazotype Material
Kalle & Co. *BP* 853,020 (Germany 18 Jan 1958)

A light-sensitive layer contains a diazonium compoind derived from a compound of the *p*-phenylenediamine series in which the benzene ring has two alkoxy groups, each of > 2 C, in *para* position to one another, and in which both H atoms of one of the amino groups are substituted by Alk or a heterocyclic ring of which the N of the amino group forms a part, the other amino group being diazotised, e.g. $ZnCl_2$ double salt of 4-diethylamino-2,5-di-n-butoxybenzene-1-diazonium chloride. It has good light sensitivity and yields by the semiwet process black prints rich in contrast of good fastness to light. C.O.C.

Colour Photography
International Polaroid Co.
BP 853,478 (U.S.A. 13 Feb 1956)
BP 853,480–1 (U.S.A. 13 Feb 1956)

Improved diffusion transfer-reversal process using an integral, multilayer, negative photosensitive element.

BP 853,479 (U.S.A. 13 Feb 1956)

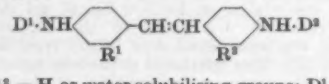
Describes azo dye developers for use in the above process.

BP 853,482 (U.S.A. 13 Feb 1956)

Describes amino aralkyldihydroxybenzenes, their *O*-acyl derivatives and their use as intermediates in making the azo dyes of *BP* 853,479 (above) and as developers in the process described in *BP* 853,478 and 853,480–1 (above). C.O.C.

Bis - (s - triazin - 2 - ylamino)stilbenes in Colour Photography
Kodak *BP* 852,066–9 (U.S.A. 12 Oct 1955)
BP 852,074–5 (U.S.A. 12 Oct 1955)

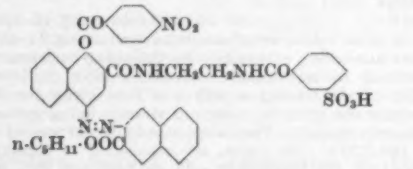
Compounds of formula—



(R^1 and R^2 = H or water solubilising groups; D^1 and D^2 = an s-triazin-2-yl nucleus), e.g. 4,4′-bis[4-(3-sulphanilino)-6-amino-s-triazin-2-ylamino]stilbene-2,2′-disulphonic acid, much increase the sensitising power of many cyanine dyes. Their presence also inhibits many colour couplers from staining or fogging photographic emulsions. C.O.C.

Colour Photography
Kodak *BP* 853,922 (29 Dec 1955)

Modification of *BP* 783,730 (J.S.D.C., **73** (1957) 573). The coloured colour coupler used has an auxochrome capable of alteration to change the colour of the coupler. This auxochrome is altered before, during, or after coupling so that the uncoupled residue left to serve as a coloured masking image has the changed colour. An example is the yellow—



which when treated with an aqueous alkaline solution loses its nitrobenzoyl group and becomes magenta. C.O.C.

Recording Media Sensitive to Ultraviolet Radiation but not to Light
International Business Machines Corpn.
USP 2,927,025 (23 Oct 1956)

The leucocyanides of triarylmethane dyes in acid solution develop brilliant colours on exposure to ultraviolet radiation and these colours do not fade in darkness. 2-Methyl-4,5,6,7-tetrachlorobenzotriazole is transparent to ultraviolet radiation but is opaque to radiation of 2800–3400 A. Thus a mixture of the two applied as a film to a base is unaffected on exposure to light but becomes brilliantly coloured on exposure to ultraviolet radiation. C.O.C.

Mordants for Acidic Organic Compounds (III p. 123)
Non-staining Triarylmethane Colour Base and Printing Therewith (IV p. 129)
Wrinkle Finish Composition (V p. 132)
Dyeing Textile Articles to give them Areas of Gradually Varying Colours (VIII p. 139)
Rendering Polyethylene Surfaces Receptive to Printing Ink (XIII p. 144)
Coated Polyethylene showing good Receptivity for Printing Inks (XIII p. 144)

X— SIZING AND FINISHING

Drying Problems and the Use of the Perforated Drum Dryer
W. Friedel *Melliand Textilber.*, **41** (Oct 1960) 1264–1266

Efficiency of drying depends on temperature and speed of air current. Highest air speed is obtained by arranging drums in one row (not staggered). Reduced drying times permit use of higher temperature as no fibre damage occurs. Perforated drum dryers for loose material have been successfully used for several years. Machines with greater working width (e.g. for carpets) are now available. Heaters and fans are arranged above and below the drums. More heaters can thereby be accommodated and the machine can be used for high-temperature processes. W.M.

Adhesion of Rubber to Textiles
R. Guyot *Rev. gén. caoutchouc*, **35** (1958) 1349–1354
Chem. Abs., **54** (10 Nov 1960) 23397

Discussion of static and especially dynamic evaluation of rubber–textile laminates and of cords treated with isocyanates, proteins, synthetic resins and/or latices, and of the factors influencing each type of treatment. Optimum ratios of HCHO to resorcinol (3), resin to natural latex (25–35%), latex particle size, total pickup (4·0–4·5%), and use of vulcanising agents are considered. Use of a positive latex is not recommended. Complete condensation of the resin, moderate drying temperatures, and 3·5–4·0% residual moisture for rayon are recommended, with a reducing atmosphere for vinyl pyridine latex and an oxidising atmosphere for other latices. Effects of the chemical and mechanical properties of the textile are considered. C.O.C.

Polymerisation and Condensation of *N*-Methylolacrylamide in Cotton Fabric
H. Kamogama *J. Soc. Textile Cellulose Ind. Japan*, **16** (July 1960) 549–555

Study of the polymerisation and condensation of *N*-methylolacrylamide in cotton fabric using either polymerisation catalysts, e.g. H_2O_2, or acid condensation catalysts, e.g. NH_4Cl, showed—(1) Drying after impregnation and before curing is unnecessary but when direct curing is used care must be taken to prevent overcuring. (2) Crease recovery and flat abrasion resistance of treated fabric increase with resin content whereas tear strength decreases. (3) Resin content and crease recovery value increases directly with the amount of catalyst used. (4) Acid catalysts give the better crease recovery values. (5) The treated fabrics have high resistance to boiling alkali washing. C.O.C.

The Crease-resisting Process: Additives
J. T. Marsh *Text. Manuf.*, **86** (Nov 1960) 449–453

The well-known tendency of crease-proofing amino resins to increase fabric stiffness and reduce extensibility, tensile and tear strength, and abrasion resistance can, to some extent, be corrected by the addition of conventional softeners, polymer emulsions or silicone resins. None of these additives penetrates the fibre or plasticises the amino resin. Softeners function by reducing inter-fibre friction and most contain a long unbranched aliphatic chain attached to a polar group, e.g. $-OSO_3Na$, $-NR_3Cl$ or $-(OCH_2CH_2)_nOH$, which determines the substantivity to different fibres. Softeners do not impart fullness but only smoothness of handle. Alternatively the long alkyl chain may be attached to the amino resin intermediate, as in an alkylmelamine. Softeners improve tear strength by assisting in the distribution of applied loads and they reduce needle damage in high-speed stitching. Polymer emulsions generally improve abrasion resistance, and tensile and tear strength, and elastic polymers improve crease recovery. Polymers of vinyl chloride and vinyl acetate are too hard to be of value, but polyalkylacrylates and butadiene–acrylonitrile copolymers are effective,

although the last named tends to yellow in the chlorine-retention test. Polymeric additives lead to greater dust pick-up and soil pick-up during laundering; polyacrylates are worse than polyethylene in this respect. They are most effective in improving the mechanical properties of loosely woven fabrics. Polydimethylsiloxane of high mol. wt. develops water repellence at 1% treatment levels and polymethylsiloxane imparts poor handle, but blends of these two polymers at $< 0{\cdot}5\%$ on the weight of fabric give good crease recovery, especially in the wet state, and good handle. Generally, polysiloxanes are best for improving tear strength but not tensile strength or abrasion resistance, but are to be preferred to polymer emulsions for tightly woven fabrics. E.C.

Resin Finishing of Cellulosic Fibres
M. Yajima and K. Arakawa

I— Effect of Various Resins on Cellulosic Fibres
J. Soc. Textile Cellulose Ind. Japan, **16** (June 1960) 500–504

Melamine–formaldehyde resins are better for use on viscose rayon than are the urea–formaldehyde resins, while ethylene urea resins are unsuitable. As the strength of the resin bath increases, the wet strength of the treated fibres increases, but dry and wet elongation, knot strength, and water imbibition decrease. Resistance to flexing is greatest with the melamine-resin-finished fibre.

II— Resin Finishing of Viscose Rayon of Various Deniers *Ibid.*, 505–506

When a melamine resin is used the amount of fixed resin increases with increase of denier. With both treated and untreated fibres dry and wet strengths and water imbibition decrease with increase of denier but dry and wet strengths decrease more with the resin-treated fibre than with the untreated.

III— Condition of the Resin Bath
Ibid., **16** (July 1960) 584–588

When applying a melamine resin to viscose rayon penetration into the fibre is retarded at $> 60°C$. The optimum padding time is 10 min. As the pH of the bath falls, the amount of resin forced on the fibre increases and water imbibition, knot strength, dry and wet elongation, and dry strength decrease. Wet strength and tensile recovery are at a maximum when the resin bath is at pH 4.

IV— Degree of Expression of the Resin Liquor and Preheating *Ibid.*, 588–590

As the degree of expression decreases, resin fixed on the fibre increases, and dry and wet strength decrease. Water imbibition is at a minimum when the degree of expression is 70%. The degree of expression has no relation to elongation or flexural rigidity of the resin-treated fibre. As the temperature of preheating decreases the fixed resin increases, wet strength and flexural rigidity increase, whereas water imbibition and knot strength decrease and tensile recovery and elongation are unaffected. Optimum preheating is 2 h at 50–60°C. C.O.C.

Shrink-resist Processes for Wool. II— Commercial Methods *Wool Sci. Rev.*, No. 18 (Oct 1960) 18–37

A detailed review of the general working conditions and control of commercial anti-felting processes, a sequel to an article appearing in *Wool. Sci. Rev.*, No. 17 (1957) 16. After general considerations have been discussed, the article describes acid wet chlorination and its modifications, alkaline wet chlorination, dry gaseous chlorination, resin treatments, and other processes (permonosulphuric acid, peroxide–copper, non-aqueous solutions of alkali, enzyme, and sodium chloride–potassium permanganate). It is concluded that the perfect process for making wool resistant to felting has still to be discovered. 25 References. M.T.

Stiffness of Silk Fibres. IX— Softening Raw Silk with Mixtures of Urea and other Compounds
H. Takagi *J. Soc. Textile Cellulose Ind. Japan*, **16** (June 1960) 496–499

Mixtures of urea and other compounds used for softening raw silk gave the following results: (1) Urea and a higher alcohol, using the alcohol sulphate as emulsifying agent, had a comparatively strong action. (2) After 70°C is reached the take up and the effect become stronger. The influence of the chain-length of the alcohol and of pH were not clear. (3) The treated silk becomes more like untreated silk with the passage of time. (4) Application of urea and then the other agent gives little or no softening action. (5) A mixture of urea and Na cyclohexylsulphamate has a fairly strong softening action which becomes strong if a higher alcohol is added to the mixture. A mixture of urea and KCNS also has a comparatively strong softening action. C.O.C.

Studies on Rabbit Fibres (XXXII, XXXIII)— Carrotting Reactions
S. Ikada *J. Soc. Textile Cellulose Ind. Japan*, **16** (July 1960) 529–538

When rabbit hair is carrotted with a mixture of $Hg(NO_3)_2$, HNO_3, and $NaNO_2$ there is no apparent increase in the glycine, alanine, proline, valine, leucine, methionine, aspartic acid, and glutamic acid components of the fibres. There is visible increase in the amounts of phenylalanine, histidine, serine, and threonine and greater increase of tyrosine, tryptophan, lysine, hydroxylysine, and arginine, while that of cystine is the largest. C.O.C.

Developments designed to Combat the Pilling Problem
A. J. Hall *Text. Rec.*, **78** (Jan 1961) 57–59

A brief review of methods of measuring pilling tendency, theoretical aspects of pill formation, and special finishing treatments to inhibit the formation of pills on woven and knitted fabrics. M.T.

PATENTS

Fire- and Vesicant-vapour-resistant Finish
U.S. Secretary of War *USP* 2,926,097 (3 Nov 1944)

The fabric is treated with a composition containing a chloroamide, a binder, and ZnO, e.g. a mixture of acetylene tetrachloride (900), sorbitan monolaurate (22·5), chlorinated paraffin wax (90), *NN'*-2,2',4,4',6,6'-octachloro-sym-diphenylurea, and ZnO (907) to give a pick-up, after drying, of 35–40%. C.O.C.

Water-repellent, Crease-resistant Finish
N. J. Brown *BP* 849,163 (21 Jan 1957)

Woven cloth is immersed for 2–5 s at 120–140°F in a solution containing a vinyl chloride–vinyl acetate copolymer, a volatile solvent and a plasticiser and is then dried at 140–160°F. The solution does not penetrate the individual fibres but coats each of them. The product looks the same on both sides, is soft and flexible, water-repellent, non-crushing and non-creasing, and has a soft handle. C.O.C.

Water-repellent Finish
ICI *BP* 848,336 (1 Feb 1957)

Textiles are given excellent water repellency by treatment in an organic solvent solution of an organopolysiloxane fluid and a zirconic ester (ester groups are aliphatic or aromatic of < 19 C), e.g. tetrabutyl zirconate. The solution contains 0·25–5·0% of the ester. The treated textiles are then heat cured, e.g. for 10 min at 100°C or for 7 min at 150°C. C.O.C.

Water-repellent and Water-borne Stain-resistant Finish
Gagliardi Research Corpn. *USP* 2,926,062 (5 Mar 1956)

Fibres of all types are treated with an emulsion containing a water-repellent wax free from glycerol and containing at least one reactive OH group, dispersed in an aqueous solution of a thermo-setting, chlorine-resistant, heterocyclic nitrogenous cross-linking agent which at $<$ 30°C neither reacts with the wax nor polymerises. The emulsifying agent is a water-soluble anionic compound capable of being rendered inactive at high temperatures. Thus carnauba wax is emulsified in water with triethanolamine dodecyl benzene sulphonate as the emulsifier. After cooling, bis(hydroxymethyl)ethylene urea is added. Cloth and wallpaper treated with this emulsion and cured at 50°C for 1 h are water-repellent and highly resistant to water-borne stains. C.O.C.

Water-repellent Finish and Coating for Textiles
J. B. Rust and W. B. Canfield *USP* 2,923,698 (4 Nov 1952)

A higher fatty acid amide is treated with a formaldehyde-yielding substance and then with a secondary amine to yield a tertiary amine reaction product. This is then converted into a water-soluble or water-dispersible non-quaternary tertiary amine salt. This in aqueous suspension is useful for impregnating or coating textiles to yield after baking a durable water-repellent finish resistant to washing. 19 Examples. C.O.C.

Wet Crease-shedding and Crease-resisting Finish for Cellulose Fabrics
Courtaulds *BP* 852,962 (12 Nov 1957)

Fabric containing cellulose fibres is treated in absence of tension with a diamine, stretched, and the diamine washed out and the fabric dried before the tension is released. Thus a spun viscose rayon fabric was treated in open width and in absence of tension with ethylene diamine at room temperature for 2–3 min. It was then stretched on a frame to 140% of its wet, relaxed area and washed with hot water until free from diamine. After drying on the same frame and to the same area the fabric was wet crease-shedding and wet crease-resisting. C.O.C.

Imparting Increased Recovery from Creasing to Cyanoethylated Cotton Fabric with a Methylol Melamine Resin
American Cyanamid Co. *USP* 2,923,597 (31 Mar 1955)

When cyanoethylated cotton is given a crease-shedding finish with a methylol melamine resin there is little or no loss in strength and markedly improved tear resistance. C.O.C.

Raising the Sticking and Safe-ironing Temperatures of Cellulose Triacetate Fibres
BrC *BP* 852,729 (28 Mar 1956)

Treatment with carboxylic esters, ethers or ether-carboxylic esters of polyhydric alcohols is used. Thus treatment with 3–10% aqueous diethylene glycol diacetate at 95–100°C for 1–3 h raises the safe ironing temperature of cellulose triacetate fibres from 180 to $\lessdot$ 220°C.

BP 852,730 (29 Mar 1956)

The fibres are treated with a swelling agent at $\lessdot$ 100°C. Thus cellulose triacetate cloth is impregnated with 60% of its weight of an aqueous solution containing by volume 25% each of benzyl and isopropyl alcohols and then is treated in a cottage steamer for 7 min with steam of pressure 5 lb/in² gauge, and then washed and dried. Its safe-ironing temperature is raised from 180 to $\lessdot$ 210°C. C.O.C.

Setting of Keratinous Fibres
Société Monsavon l'Oreal
BP 849,045 (France 20 May 1957)

The fibres are treated, before or after being placed in the desired form, with an aqueous solution at pH 9–10 of an alkali-metal sulphite or bisulphite (1 mol.) and Na and/or K borohydride (1–20 ml). The strength of the solution is such that the active H available is $\gtrdot$ 2 g/100 ml. After being treated with the solution for $\lessdot$ 15 min, the fibres are rinsed, fixed and dried. C.O.C.

Reducing the Felting Power of Wool
U.S. Secretary of Agriculture
USP 2,925,317 (18 Sept 1956)

When wool is treated with a preformed polyalkyleneimine its felting power is much reduced. The maximum effect is obtained by treatment at pH 9–11 followed by heat curing. Thus woollen cloth was impregnated with 120% of its dry weight of a 5% aqueous solution of polyethylene imine (mol. wt. 20,000–30,000) at pH 11·3 and was then baked for 60 min at 120°C. The treated cloth showed no area shrinkage on being violently agitated in 0·5% soap solution at 40°C with a liquor ratio of 50:1 whereas the untreated cloth shrank 36% in area. Treated cloth which had been washed for 2 h in running water at 55°C also showed no shrinkage when given the above test. C.O.C.

Irradiation of Synthetic Linear Polymerisation or Polycondensation Products
N.V. Onderzoekinginstituut Research
BP 852,954 (Holland 16 June 1956)

Fibres, sheeting, etc. of the polymers or polycondensates are slightly swollen so that they contain $\gtrdot$ 25% by wt. of the swelling agent and are then subjected to ionising radiation. The resultant decrease in shrinking tension exceeds that obtained by swelling or irradiation alone. Thus 840-denier polyhexamethylene adipate yarn was wound on an elastic spool and immersed in water at 20°C. After 24 h it was unwound and led around two non-parallel rollers along a helical path at 4 m/min under a tension of 50 g. Between the rollers it was irradiated with electrons, the irradiation dose amounting to 5×10^6 rep. It was then dried in absence of tension and conditioned at 20°C and 65% r.h. The shrinking tension at 145°C of the treated yarn was less than half that of the untreated yarn. C.O.C.

Durable Antistatic Finish on Hydrophobic Textiles
Onyx Oil & Chemical Co.
BP 854,381 (U.S.A. 13 Apr 1956)

The textiles are impregnated with (*a*) a water-soluble linear basic polyamide having recurring units—

$$\left[(CH_2)_n\underset{\displaystyle R^1}{N}\!\!-\!\!(CH_2)_n NHOCR^2CONH\right]_k$$

(R^1 = N, Alk or aralkyl, R^2 = covalent bond or subst. or unsubst. aliphatic, aromatic or alkylaromatic; k = 1–4; n = 2–8) and (*b*) a diester alkylating agent capable of cross-linking and insolubilising the polyamide. They are then dried and baked. C.O.C.

Silken Finish on Polyester Fabrics
DuP *BP* 847,147 (U.S.A. 12 Dec 1956)

Polyester fabrics are made to resemble silk fabrics by being hot pressed so that the cross-sections of the fibres are made to resemble those of natural degummed silk. They are then heat-set and treated with alkali so that they lose $\lessdot$ 5% by weight. C.O.C.

Anti-soiling and Anti-static Finish for Hydrophobic Fibres and Films
ICI *BP* 850,169 (30 Jan 1956)

The fibre or film is first soaked in aqueous caustic alkali and then with a lipophilic agent. When $\lessdot$ 0·2% by wt. of the lipophilic agent is absorbed into the surface, the fibre or film is heated until it is saturated by the agent. Thus white fabric of worsted-spun Terylene is soaked for 12 h in 5% aq. NaOH at room temperature. It is then squeezed off and, while still wet, padded for 20 min with a 1% aqueous solution of Cirrasol 8F 200 so that it picks up 0·7% of its weight of Cirrasol. Finally it is steamed for 20 min with steam of pressure 20 lb/in². This yields an excellent soil-resistant finish. C.O.C.

Heat Stabilising of Polyester Yarns
Fabric Research Laboratories
USP 2,926,065 (18 Apr 1956)

Yarn of polyethylene terephthalate is heated at $>$ 340°F while under tension to prevent it shrinking. Preferably the tension should be such as to stretch the yarn by $<$ 50%. It is then heated to 340–400°F while being allowed to shrink, only such tension being maintained as is necessary to prevent snarl formation. C.O.C.

Treating Fabric with Powder—Laminating Fabrics
H. P. Barns *BP* 850,791 (11 Oct 1955)

A water-insoluble polymer is sprayed or dusted on to the fabric. If necessary heat and/or pressure may be used to cause the powder to adhere to the fibres. The polymer may be a powder or if thermoplastic may be sprayed on while molten. A second layer of fabric may be placed upon the treated fabric and bonded to it by heat and pressure but without causing the particles to coalesce into a continuous film or large aggregates. C.O.C.

Fire-resistant Bonded Fibre Fabric
Courtaulds *BP* 849,114 (23 Jan 1957)

A carded lap containing cellulose acetate ($\gtrdot$ 50% by wt.) and alginate fibres ($\lessdot$ 50) and other fibres (0–10%) is bonded together by rendering the acetate adhesive. Presence of the alginate fibres renders the resulting fabric markedly fire-resistant. The fire-resistance of the cellulose acetate may be improved by impregnating it before or after formation of the fabric with a fire-resisting agent. C.O.C.

Air-impervious Coated Fabric
De Bell & Richardson *BP* 848,811 (U.S.A. 30 Dec 1955)

Cloth is woven from multifilament polyethylene terephthalate yarn of 7–9 g/denier and which, if fully shrunk while relaxed at 230–275°F and then cooled, has elongation at break of 12% and shrinkage of $\gtrdot$ 4% when kept at 230°F for 4 h and $\gtrdot$ 6·25% when kept at 275°C for 4 h. It is then coated on both sides first with methylene di-isocyanate and then with chlorosulphonated polyethylene. Finally the coating is cured by baking. C.O.C.

Artificial Fur
Diamond Alkali Co. *USP* 2,925,359 (16 Feb 1960)

Two fabrics are woven in one of which the pile simulates the guard hairs and in the other the underfur of natural furs. The warp consists of fibres which can be removed by

chemical or physical treatment which does not affect the pile fibres. The fabrics are then cut into long strips, the underfur fabric being made into narrower strips than the guard hair fabric. They are then assembled so that their width is vertical. They are placed alternatively in the assembly, e.g. an underfur strip is sandwiched between layers of guard hair strip. The assembly is laminated together and a backing applied to the lower of the edges of the strips. The warp threads are then dissolved out or otherwise removed. This leaves an artificial fur having long guard hairs and shorter underfur anchored to the backing. C.O.C.

Reagents for Crease-resist Processing (III p. 123)
Flame-retardant Coating Compositions (V p. 133)
A Study of "Static" in Relation to the Soiling of Garments in Wear (VI p. 133)
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Effect of High-energy Radiation on Wool Keratin and Polyamide and Polyester Fibres (VI p. 134)
Cellulose Anthranilate Fibres (VI p. 135)
Wrinkle Finish Printing (IX p. 139)

XI— PAPER AND OTHER CELLULOSIC PRODUCTS

White Pigments and White Paperboard

W. R. Willets *Pulp Paper Mag. Can.*, **61** (C) T 155–T 160 *Chem. Abs.*, **54** (10 Nov 1960) 23322

In selecting a pigment, attention has to be paid to uniformity, brightness, whiteness, hiding power, particle size, freedom from grit, ink affinity, ability to produce satisfactory gloss, ease of dispersion, chemical inertness, and price. Final selection necessitates compromise among all these. The importance of proper choice of pigments for calendering, coating and beater addition is indicated by reference to work on varnish holdout, ink receptivity, and adhesive requirements. The application of coatings to various types of board is discussed. C.O.C.

Ion-exchange Celluloses for Chromatographic Separations

J. D. Guthrie and A. L. Bullock *Ind. Eng. Chem.*, **52** (Nov 1960) 935–937

The low capacity of ion-exchange celluloses, usually prepared by introducing acidic or basic groups into the cellulose chain, has been improved by treatment with cross-linking agents before chemical modification. The fibrous structure is retained during the latter process. Cross-linking agents used included di(2-sulphatoethyl)-amine, 1,4-disulphatobutane, 1,3-dichloro-2-propanol, divinylsulphone, and formaldehyde. Thus cotton linters were treated with acidified 36% formaldehyde soln., washed under alkaline and neutral conditions, and carboxymethylated in 30% mono-chloroacetic acid, excess of acid was removed, and the Na salt formed by immersion in 40% NaOH for 15 min. Carboxymethylation was repeated twice, the resultant chemically modified cellulose retaining its fibrous structure, whereas non-cross-linked sodium carboxymethylcellulose was water-soluble. The cation exchange value increased to 1·9 meq. per g. Cross-linking and chemical modification may be simultaneously achieved by treatment with dichloroacetic acid, followed by hot 50% NaOH. The preparation of cross-linked diethylaminoethylcellulose is also described. Ion-exchange celluloses are valuable for separation of amino acids, proteins, enzymes, etc. F.J.

PATENTS

Paper containing a Fluorescent Brightening Agent

FBy *USP* 2,924,549 (U.S.A. 15 Dec 1955)

Use of a white siliceous pigment and a fluorescent brightening agent having bluish fluorescence enables reduction in the amount of white pigment necessary to obtain the desired opacity and whiteness. C.O.C.

Modified Photographic Paper

Kodak *BP* 858,060 (U.S.A. 26 Apr 1956)

Wet-stiffness is doubled, lateral shrinkage reduced to $< 1\%$, and change in thickness on wetting to $< 15\%$ when a partially acetylated paper is wetted with a water-miscible liquid, e.g. water, alcohol, acetone, and dried at $> 200°F$ under pressure against a polished metal surface. R.A.

Wrinkle Finish Printing (IX p. 139)

XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Physical Properties of Leather Finish Films

W. O. Nutt *J. Soc. Leather Trades Chem.*, **44** (Nov 1960) 519–524

The performance of leather finishes may be related to the constitution and properties of the film-forming material. Tests, based upon measurements of adhesion and dry-rub resistance, show that changes of properties may be obtained in a predetermined way once it is understood how the component materials affect the formation of the finish film, and the effect of film formation on film performance is known. Examples discussed relate to the modification of acrylic resin finishes by addition of casein and waxes, and by alteration to the charge by reversal or intensification. J.W.D.

The Temperature Dependence of the Breaking of Hydrogen Bonds of Collagen by Urea

K. H. Gustavson *J. Amer. Leather Chem. Assocn.*, **55** (Oct 1960) 564–584

The solubilisation of hide collagen by concentrated soln. of urea (4–8 M) is highly temperature-dependent. Little degradation occurs at temperatures below 30°C, but marked degradation of the skin structure takes place above 30–35°C. It is shown that the action of the urea is to break the hydrogen bonds of the collagen, resulting in solubilisation. The bearings of these findings on the concepts of the mechanisms of the major tannages are outlined. P.B.S.

Structure and Chemical Constituents of Collagen

N. T. Crosby *J. Soc. Leather Trades Chem.*, **44** (Dec 1960) 551–561

An essay, awarded the Donald Burton Prize for 1960, which reviews present knowledge of the amino-acid composition of collagen, peptide theory of protein structure, distribution of amino-acids along chains, and physical and chemical studies on soluble collagens. The structure of collagen, determined by X-ray diffraction and electron microscopy, is reviewed briefly. F.J.

PATENTS

Treating Leather with Castor Oil and Polyisocyanates

ICI *BP* 857,120 (31 Aug 1959)

Treating leather with castor oil and then with a polyisocyanate makes it water-repellent, while leaving it flexible and porous. Resistance to wear is increased and the leather dries very rapidly after being wetted with water. In addition, tanning materials and colorants present are given increased wet fastness. C.O.C.

Rendering Leather Water-repellent

Wacker-Chemie *BP* 858,024 (Germany 21 Nov 1956)

The leather is treated with a compound of formula—

$$R_nSiO_{(4-n)/2}$$

(R = monovalent hydrocarbon; n average value 1·0–2·9) and an aqueous solution of a zirconyl salt. The two treatments may be in that order or simultaneous. As the polysiloxane cures only slowly it is possible to dress or finish the treated leather with aqueous solutions before the final water repellent-finish is produced. C.O.C.

Dyeing Suède Pigskins (VIII p. 137)
Artificial Fur (X p. 142)

XIII— RUBBER; RESINS; PLASTICS

Gelation and Formulation Theory of Ester Resin

C. W. Johnston *Off. Dig. Fed. Soc. Paint Technol.*, **32** (Oct 1960) 1327–1346

An account of the theory of gelation based on average functionality of the acids and polyols present. A simplified formula, based on the original work of Flory, is developed for the calculation of the mol. wt. of the resins produced

and for predicting gelation. The development of new resins and the prediction of gelation acid values is facilitated.
J.W.D.

Use of Resins in Bonded Fabrics
British Plastics, 33 (Dec 1960) 564–567
A survey of present manufacturing processes, with six photographs, indicating also the present end-uses.
J.W.D.

PATENTS

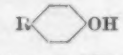
Antioxidants giving Protection against Ozone Cracking
United States Rubber Co.
BP 856,286 (U.S.A. 12 Nov 1957)
N - Alkyl - *N'* - (*p* - alkylphenol) - phenylenediamines impart excellent protection to rubber vulcanisates against ozone cracking as well as bulk oxidation. C.O.C.

Molten Metal Bath for Curing Plastics, Rubber, etc.
Okonite Co. *BP* 857,323 (U.S.A. 30 June 1958)
Zones of different temperatures are maintained within an open, static body of molten metal, the zones being so placed that material passing through the bath has its temperature progressively and gradually raised. This enables the material to be very rapidly heated and also to be subjected to any desired hydrostatic pressure at any time during curing. C.O.C.

Stabilised Acrylonitrile Polymers
Chemstrand Corpn. *BP* 859,508 (U.S.A. 17 Dec 1956)
A mixture of paraformaldehyde and triethyl, tributyl, tris(2-ethylhexyl), trihexyl or triso-octyl phosphite gives excellent colour stability to acrylonitrile polymers when they are heated or to their solutions when kept for long periods.
BP 859,509 (U.S.A. 17 Dec 1956)
A mixture of a compound of formula—

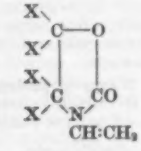
$$(RSOO)_nM$$

(R = alkanol or acyl of 1–3 C or Ar of 6–8 C; $n = 1$ or 2; M = Na, K or Zn) e.g. Zn formaldehyde sulphoxylate and an organic phosphite, a sulphonic acid, e.g. phenol disulphonic acid or benzene phosphoric or benzene phosphorous acid, is used.
BP 859,510 (U.S.A. 17 Dec 1956)
A mixture of (*a*) HCHO and a sulphoxylate of the above formula or of (*b*) acetic acid or acetic anhydride plus (*a*) or of (*c*) HCHO, acetic anhydride and a compound of formula—

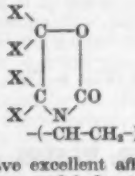


(R = H, methoxy, ethoxy or OH) is used. C.O.C.

N-Vinyl-X-alkyl-2-oxazolidines and their Polymers
Dow Chemical Co. *BP* 851,773 (U.S.A. 14 Nov 1957)
Compounds of formula—



(X = same or different H or Alk of < 5 C, one X at least must be Alk), e.g. *N*-vinyl-5-methyl-2-oxazolidone, readily form polymers having the recurring group—



These polymers have excellent affinity for a wide range of dyes and so are useful for incorporation in many synthetic polymers which otherwise are difficult to dye.
C.O.C.

Avoiding Discoloration by Iron in Plastics
Argus Chemical Corpn. *BP* 857,382 (U.S.A. 11 Oct 1957)
Addition of very small amounts of an organic polyhydroxy compound (< 2 alcoholic OH groups) prevents discoloration of plastics by iron, e.g. a vinyl chloride–vinyl acetate (87:13) copolymer is given good protection by addition of 6% of its weight of pentaerythritol. C.O.C.

Vinyl and Vinylidene Polymers Pigmented with Carbon Black (C.I. Pigment Black 6 and 7)
Crylor *BP* 859,292 (France 5 Dec 1957)
A vinyl or vinylidene monomer is adsorbed into Carbon Black in absence of any resinous polymer and is then polymerised. The products can be used as moulding powders or extruded to form, e.g., filaments. C.O.C.

Contrast Colour Embossed Plastics
Dymo *USP* 2,925,625 (22 Oct 1958)
Some rigid plastic sheet materials, especially straight unplasticised polyvinyl chloride and polyvinyl chloride–polyvinyl acetate copolymers, develop a highly contrasting or opaque colour value in regions subjected to cold flow, i.e. viscoelastic deformation. This principle is made use of, e.g. by embossing, to produce designs of sharply contrasting colour values upon sheets of such plastics. C.O.C.

Rendering Polyethylene Surfaces Receptive to Printing Ink
Celanese Corpn. of America *USP* 2,923,964 (22 Dec 1953)
The surfaces are subject to a high-voltage electrical discharge, more particularly to a diffuse corona discharge.
C.O.C.

Coated Polyethylene showing good Receptivity for Printing Inks
Celanese Corpn. of America *USP* 2,925,354 (16 Feb 1960)
Treatment with a mixture of chlorosulphonated polyethylene and cyclised rubber and then baking for a short while results in a polyethylene surface to which printing inks show good adhesion. C.O.C.

Decorative Laminated Plastic Material
Goodyear Tire & Rubber Co.
BP 854,929 (U.S.A. 6 Mar 1958)
A thin transparent thermoplastic resin film having one side pitted so that it is duller than the other side is calendered. An opaque backing sheet of thermoplastic resin of thicker gauge and lower resin content than the first film is superimposed against the duller side of the sheet after both the sheet and film have been individually heated. They are then caused to adhere together by applying hot pressure. The duller surface of the sheet may be coloured or have a design printed on it. C.O.C.

Flameproof Composition for Application to Plastics and Resins
Diamond Alkali Co. *USP* 2,924,532 (9 Sept 1957)
The composition consists essentially of a mixture of a highly chlorinated high-mol.-wt. aliphatic material and an organic antimonyl compound. It is preferable that either the composition or the plastic contains a stabiliser for chlorinated aliphatic materials. Thus a mixture of potassium antimonyl tartrate (15), chlorinated paraffin wax (70% combined Cl) (15), 50% solution of benzoyl peroxide in tricresyl phosphate (4) and Co naphthenate (0·1) incorporated in a polyester resin (70) renders it flameproof. C.O.C.

Decorative Sheet Material
Congoleum–Nairn *USP* 2,926,100 (24 Dec 1956)
An impregnated felt backing and particles of thermoplastic synthetic resinous composition are passed simultaneously through the nip of two calender bowls so as to form the composition into a sheet and bond it to the felt in one single passage through the nip. C.O.C.

Organic Protective Coatings (V p. 131)
Coating Fabrics with P.V.C. (VI p. 135)
Stabilising Polyamides (VI p. 135)
Wrinkle Finish Printing (IX p. 139)
Adhesion of Rubber to Textiles (X p. 140)
Irradiation of Synthetic Linear Polymerisation or Polycondensation Products (X p. 142)
Physical Properties of Leather Finish Films (XII p. 143)
Infrared Spectra of Crystalline Poly-*p*-fluorostyrene (XIV p. 147)
Infrared Spectrum of Some Deuterated Crystalline Polystyrenes. I—Isotactic Poly-α*d*-styrene (XIV p. 147)

XIV— ANALYSIS; TESTING; APPARATUS

Amended Method for Determination of Wetting Power of Surfactants

R. Schiffner *Melliand Textilber.*, **41** (Oct 1960) 1275–1277

A disc-shaped sample is pressed under the surface of the liquid and the time required for the sample to sink is measured. W.M.

Improved Method of Determining Wetting-out Properties of Surface-active Agents

E. Schiffner

Faserforsch. und Textiltech., **11** (Oct 1960) 500–502

A 35-mm-diameter sample of cotton wool is fixed vertically in a holder so that it can be immersed to a fixed depth in a solution of the surface-active agent. The time required for wetting out is then determined. The apparatus is described and results are compared with those obtained by other methods. W.R.M.

Detection and Estimation of Polyoxyethylene Glycol in Non-ionic Surfactants by Paper Chromatography

M. E. Ginn, C. L. Church, Jr., and J. C. Harris

Anal. Chem., **33** (Jan 1961) 143–145

A water- and acetic acid-saturated solution of 1-butanol is used as developing solvent in ascending paper chromatography. After developing for 5 h, the chromatographed spots are detected by spraying with a modified Dragendorff reagent. Measurement of spot area is found to give quantitative results accurate to the nearest 1%. P.B.S.

Absorptivity Recorder

D. D. Tunnicliff and C. Hawes

J. Opt. Soc. Amer., **50** (Nov 1960) 1039–1042

An accessory for the Cary recording spectrophotometers is described, which converts and records linear optical density measurements on a semilog scale as extinction coefficients against wavelength. By the use of a range of cell lengths and concentrations a complete absorption spectrum can be recorded in the same absorption units throughout the wavelength range of the spectrophotometer. The extinction coefficients are claimed to be accurate to within 2%. With suitable modifications the unit could be used with most spectrophotometers which record spectra on a linear absorbance scale. E.COATES

Sensitisation of Photoconductivity in Glass by Dyes

R. C. Nelson *J. Opt. Soc. Amer.*, **50** (Oct 1960) 1029

It has been noted that films of some dyes on Pyrex sensitise the volume conductivity of the glass. The dyes which are effective are also good photographic sensitisers, while those which are not effective are poor photographic sensitisers. A cell for the measurement of the effect is described and the results are discussed. An electron-transfer process seems most likely and its implications are considered. Processes of this sort may be of importance in photobiology. E.COATES

Calcein Blue— a Metalfluorochromic Indicator for Chelatometric Titrations

D. H. Wilkins *Talanta*, **4** (1960) 182–184

Chem. Abs., **54** (10 Nov 1960) 22137

Calcein Blue (a condensate of 4-methylumbelliferone, HCHO, and iminodiacetic acid) can be used for the chelatometric detection of metal ions which form highly coloured complexes with chelatometric reagents. C.O.C.

Absorption Spectra of Azo Dyes. II— Spectra of Derivatives of 4-Formyl-, 4-Nitro- and 4-(2-Nitrovinyl)azobenzene

L. Skulski and T. Urbański

Roczniki Chem., **34** (1960) 443–455 (*English summary*)

Chem. Abs., **54** (10 Nov 1960) 21997

Visible and near ultraviolet absorption spectra of the monoazo dyes p-$YC_6H_4N{:}NAr$ (Y = CHO, NO_2, $CH{:}CHNO_2$, H, $CH{:}CH_2$) are described and discussed. C.O.C.

Characterising the Purity of Water-soluble Dyes

J. Mýl and Z. Šolc *Chem. prûmysl.*, **10** (1960) 290–292

Chem. Abs., **54** (10 Nov 1960) 23335

An attempt to find a criterion of the purity of dyes of mol. wt. 350–500, especially in presence of electrolytes, e.g. Na acetate. The dyes used were obtained by coupling pyrazolone with orthanilic acid (dye I), metanilic acid (II), sulphanilic acid (III), R acid with aniline (IV), and p-nitroaniline (V). Methylene Blue (C.I. 52015) (VI) and Methyl Violet (C.I. 42535) (VII) were also used. Diffusion of the dye through a porous bar using a solution of 5×10^{-4} mol/l. was measured. Appreciable decrease in a diffusion coefficient occurs only when about 10% of a strong electrolyte is present. Measurement of the conductivity of solutions containing the same proportion of dye is a more sensitive criterion, as presence of 2–3% Na acetate become evident. The Robinson criterion (*Chem. Abs.*, **26** (1932) 1495) is limited to colloidal dyes, but can be used to judge the degree of aggregation of the pure dye in a dilute solution. II, III, and IV formed only dimers; I, V, and VII did not aggregate and only VII formed layer micelles. C.O.C.

Paper Chromatography of Water-soluble Dyes

J. C. Riemersma and F. J. M. Hestinga

Mitt. Gebiete Lebensm. u. Hyg., **51** (1960) 94–104

Chem. Abs., **54** (10 Nov 1960) 23097

Of several mixtures of an alcohol, an organic acid, and water, used as the mobile phase, best results as regards sharpness of spots, reproducibility of R_f values, and degree of separation, were obtained with t-butanol:propionic acid:water (50:12:38 by vol.) containing 0·4% HCl. As the properties of this mixture differ essentially from those of the mobile phase used by Thaler and Sommer (*Chem. Abs.*, **48** (1954) 2376), the chromatographic mechanism in the two cases is probably different. C.O.C.

Absorption Spectra of 4,4′,4″,4‴-Phthalocyaninetetrasulphonic Acid and its Metalloderivatives

H. Kobayashi, Y. Torii, and N. Fukuda

Nippon Kagaku Zasshi, **81** (1960) 694–698

Chem. Abs., **54** (10 Nov 1960) 21994

Examination of the visible and ultraviolet spectra of the monomers and associated mol. in aqueous solution, and of the monomers alone in neutral surfactant solutions, showed that the spectra resembled those of phthalocyanine (C.I. 74100) and its metallo derivatives, except for that of Fe. The degree of association, formation of micelles, etc. depended on the method of making the sols, even when using identical concentrations of dye and surfactant. C.O.C.

Chromatographic Study of Some Procion Dyes

S. R. Sivarajan and N. G. Parikh

Current Sci. (India), **28** (1959) 323–324

Chem. Abs., **54** (10 Nov 1960) 23336

0·2% Aq. soln. of the dyes were circularly chromatographed (12·5-cm-diam. Whatman No. 1 cellulose filter paper) using buffer mixtures in the range pH 6·9–12·7. R, rate of the distance travelled by the dye to that travelled by the solvent, was plotted against pH. For Procion Yellow RS (C.I. Reactive Yellow 4), Procion Brilliant Yellow 6GS (C.I. Reactive Yellow 1), and Procion Red GS (C.I. Reactive Red 5), there is a single sharp minimum for R at pH very close to 11, which is about identical for the optimum pH for maximum percentage fixation of the dyes on cotton at the same temperature (23 ± 1°C). For Procion Brilliant Red 2 BS (C.I. Reactive Red 1) and Procion Brilliant Blue RS (C.I. Reactive Blue 4), R is a minimum at at least two pH, roughly 9·0 and 11·3. These minima are not sharp and do not correspond to the pH for maximum percentage fixation. It seems that the rate of reaction between Procion dyes and cellulose is governed by several factors and is not always instantaneous, as zones of chemically combined dyes having high R values are obtained at both low and high pH. C.O.C.

Chromatography of Hydroxylated Flavenes on Carboxymethylcellulose

R. Neu *J. Chromatography*, **4** (Dec 1960) 489–493

Carboxymethylcellulose (I) has been used as chromatographic adsorbent in the preparation of flavones, flavanones and flavanols and their glycosides. Polymeric components of plant extracts are irreversibly adsorbed. Flavanols and their dihydro-deriv. and chalcones and flavanones may be separated by use of (I). Adsorption of d-catechin and l-epicatechin is reversible, in contrast to adsorption on polyamide (II). Adsorption capacity of (II) is greater than that of (I). F.J.

Determination of Concentration of an Aqueous Suspension of a Dye during Preparation of a Mother Suspension

V. K. Ponomarev and T. B. Filicheva

Khim. Volokna, (3) (1960) 62–64

Chem. Abs., **54** (10 Nov 1960) 23336

Variation of optical density of the suspension is caused

solely by variation in concentration, at least for finely dispersed suspensions. Thus, concentration of the suspension can be determined spectrophotometrically. C.O.C.

Simple and Exact Method of Assay of Food Dyes
M. Kchouk *Arch. inst. Pasteur Tunis*, **34** (1957) 517–524
Chem. Abs., **54** (10 Nov 1960) 23097

To determine whether meat bore the official stamp, small squares of it bearing the stamp under investigation and the official stamp were treated in 2 micro-kumagawas with steam to extract the dyes. After cooling and extracting the first with ether, the dyes were separated by ascending chromatography on Arche 202 paper with butanol:acetic acid:water (8:2:10) as solvent at 32°C for 20 h. Under Wood's light, a violet (R_f 0·32) and a yellow (R_f 0·93) fluorescent spot appeared in the case of the suspected stamp, while the official stamp showed three (R_f 0·09; 0·35; 0·93) red non-fluorescent spots, thus confirming that the stamp under investigation had not been applied officially. C.O.C.

Photoelectric Colorimetric Determination of Iron in Dyes
V. K. Ponomarev and T. B. Filicheva
Khim. Volokna, (3) (1960) 60–62
Chem. Abs., **54** (10 Nov 1960) 22161

A 0·2–0·3 g sample in a crucible is carefully burnt and heated in a muffle furnace for 2·0–2·5 h to oxidise all the Fe. The dry residue is cooled, treated with 5 ml conc. HCl and the acid evaporated; this is repeated twice and then a further portion of acid added, but not evaporated completely. The solution is diluted with water and filtered into a 200-ml flask, the filter paper being washed until no reaction for Cl is obtained. 2 ml 1:1 HNO_3 and 25 ml 10% NH_4SCN are added, and after thorough mixing the optical density in 10-mm cells is measured using a green filter. C.O.C.

Quantitative Analysis of Binary Fibre Mixtures Containing Courtelle (Acrylic Fibre)
W. Armfield *J. Textile Inst.*, **51** (Dec 1960) P 699–P 705

Analytical procedure is described for estimating Courtelle in blends with silk, cotton, wool, nylon, Terylene, and Fibro by dissolving the Courtelle in dimethylformamide. The method is applicable to other acrylic fibres except basified acrylics dyed with pre-metallised dyes. W.G.C.

Simple Dyeing Test to Distinguish between Courtelle, Orlon Type 42, and Standard Acrilan
K. Meldrum J.S.D.C., **77** (Jan 1961) 22

Simple Method of Determining Concentration of Chemicals in a Static Rinsing-bath
A. Freyberg
Melliand Textilber., **41** (Sept 1960) 1133–1134

The concentration of NaOH in a rinsing bath is determined as follows. If v is the volume of fresh water fed in during time t, v_1 the initial bath volume, u the concentration of the chemical at time t, u_1 the ratio of the amounts of chemical and fresh water introduced per unit time, and if v/v_1 is denoted by x, and u/u_1 by y, the equation $y = 1-e^{-x}$ applies, from which the concentration at a given time can be calculated. W.M.

Extraction of Dyes from Acrylic Fibres
D. Blackburn and K. Meldrum J.S.D.C., **77** (Jan 1961) 22

Effect of Substrate on Light Fastness
A. Schaeffer *Tinctoria*, **57** (Oct 1960) 418–421

It is suggested that light fastness depends on strength of the dye–fibre bond. If there is a very close linkage, light energy absorbed by the dye can be transferred to the fibre. This would imply parallelism between light and other (esp. wet) fastness properties. Many dyes belonging to different classes were tested, and in the majority of cases the above parallelism was confirmed. W.M.

AATCC Light Standards: New Suggestions
H. Luttringhaus
Amer. Dyestuff Rep., **49** (28 Nov 1960) 880–882

The drawbacks of using fibre standards made by blending, in various proportions, tops dyed separately with C.I. Mordant Blue 1 (C.I. 43830) and C.I. Solubilised Vat Blue 8 (C.I. 73801) are briefly outlined. Their replacement by standards of 100% worsted fabric dyed with various recipes of C.I. Solubilised Vat Blue 8 and Wool Fast Blue GLA (C.I. Acid Blue 102) is suggested. The preparation of standards claimed to be practically equivalent to the present standards 5–8, both visually and spectrophotometrically, for Fadeometer and daylight (Florida) use, is reported. S.B.D.

Perspiration Fastness of Dyes—Influence of Histidine
P. Ponchel, G. Debeauvais, G. Mazingue, and M. Van Overbèke
Bull. Inst. Text. France, (90) (Sept–Oct 1960) 39–48

A comparative study of the projected Swiss method (ISO, Lucerne, 1958) and the present E.C.E. method for the assessment of perspiration fastness. These differ in (*a*) the method of maintaining contact between dyed and undyed samples; (*b*) the addition of histidine to the test solution in the Swiss method; and (*c*) the pH of the test solution. Of these differences (*b*) is the most important. The presence of histidine increases bleeding and reduces light fastness of copper salt-treated dyeings on cellulose. Other dyes are not affected, including metal-complex dyes on wool. It is concluded that the Swiss method gives a more realistic assessment of perspiration fastness. J.C.F.

Colorfastness to Washing
V. D. Lyon
Amer. Dyestuff Rep., **49** (3 Oct 1960) P 742–P 743
Review of activities of A.A.T.C.C. Committee RA 60. M.T.

Identification of Textile Finishes
T. D. Miles
Amer. Dyestuff Rep., **49** (3 Oct 1960) P 738–P 739
Review of activities of A.A.T.C.C. Committee RA 45. M.T.

"Wash-and-Wear"—1960 Progress Report
G. M. Richardson
Amer. Dyestuff Rep., **49** (3 Oct 1960) P 734–P 738

Chairman of A.A.T.C.C. Committee RA 61 discusses the recent activities of this committee. Three working subcommittees deal, respectively, with (*a*) improved lighting and viewing procedures for testing, (*b*) methods for evaluating creases, seams, and pleats, and (*c*) development of objective methods. The principles of the Hunter Wrinklemeter, Cluett Smoothness Evaluator, and du Pont LAP method are described. M.T.

Apparatus for Measurement of Crispness of Fabrics—Application to the Measurement of Crease-recovery
J. Henno and R. Jouhet
Bull. Inst. Text. France, (90) (Sept–Oct 1960) 95–100

Describes the construction of an apparatus designed to perform the following operations—(*a*) measure force F required to close a loop of fabric; (*b*) compress loop with load of 100 g for 45 min; (*c*) allow fabric to recover for 45 min; (*d*) measure force f now required to close loop. Then $(f/F) \times 100$ is taken as a measure of the crease-recovery of the fabric. J.C.F.

Test Methods for Evaluating Shrinkproofed Fabrics
M. J. Reider *Canadian Textile J.*, **77** (11 Nov 1960) 61–66

Area shrinkage measurements were made on four all-wool fabrics of different constructions and a 75:25 wool–Acrilan fabric, both untreated and after exposure to four different shrinkproofing treatments (oxidation, halogenation, and halogen–oxidation), a 70:30 wool–Orlon fabric described as "wash-and-wear", and an all-wool, Dylanised fabric; the last two were tested as received. Each fabric was subjected to (*a*) the AATCC accelerated laundering test, (*b*) a standard hand-milling test, and (*c*) successive cycles of home laundering. The untreated control fabrics showed, in most cases, more shrinkage after the accelerated test than after 2–5 cycles of home laundering. With almost all the shrinkproofed fabrics, however treated, the home-laundering test produced as much or more shrinkage than did the accelerated test. The paradox is unexplained. With one exception (a tightly woven woollen fabric) shrinkage did not, as might have been expected, increase with test temp. The three test methods do not correlate well; the two accelerated methods are preferred, but only for their speed. J.W.D.

Rapid Identification of Cellulose Esters and Ethers
E. B. Mano and L. C. O. Cunha Lima
Anal. Chem., **32** (Dec 1960) 1772–1773

A method for the rapid identification of cellulose derivatives, making use of solubilities, colour reactions, and paper chromatography. Cellulose acetate, acetate–butyrate, nitrate, methyl-, ethyl-, hydroxyethyl-, and carboxymethyl-cellulose are investigated. All except methyl- and hydroxyethyl-cellulose, which react similarly in all tests, can be distinguished easily. No special reagents or equipment are required and the method can be applied to materials without prior purification. P.B.S.

Infrared Spectra of Crystalline Poly-*p*-fluorostyrene
M. Kobayashi, K. Nagai, and E. Nagai
Bull. Chem. Soc. Japan, **33** (Oct 1960) 1421–1427

Infrared spectra of isotactic and atactic poly-*p*-fluorostyrene were measured, to find in what respects they would reflect the characteristics of helical structure. The dichroic ratios of the bands, especially those assigned to the stretching vibrations of CH_2 groups, differed from those of polystyrene. To obtain a theoretical basis for this fact, the mol. vibrations were analysed, those associated with the side group in terms of D_{2h} symmetry and those with the skeletal chain in terms of C $(\pi/2)$ symmetry. An appendix deals with the geometrical structures of polymers with helical structure. H.H.H.

Infrared Spectrum of Some Deuterated Crystalline Polystyrenes I— Isotactic Poly-*αd*-styrene
M. Kobayashi
Bull. Chem. Soc. Japan, **33** (Oct 1960) 1416–1421

Infrared spectra of isotactic polystyrene and poly-*αd*-styrene are tabulated, and vibrational assignments are given after considerable discussion. The bands characteristic of the isotactic polymers, i.e. those appearing in the region of 1400 ~ 1180 cm^{-1} or in the vicinity of 1070 and 900 cm^{-1}, are established as associated with the CH_2 and CH groups. Remarkable changes in the spectrum on deuteration are observed in these regions, the atactic polystyrene band at 1070 cm^{-1} splitting into two σ bands at 1080 and 1048 cm^{-1}, whereas the corresponding bands of isotactic poly-*αd*-styrene appear at 1084 and 1072 cm^{-1} respectively. These bands are assumed to be associated with the characteristics of the helical polymer chain rather than with the interaction between the neighbouring mol. in the crystalline lattice, since they do not disappear even in the molten state. H.H.H.

The Ultracentrifuge
S. Dagley *J. Roy. Inst. Chem.*, **84** (Nov 1960) 382–389

A simple account of the working of the ultracentrifuge, followed by some examples of its use in the biochemistry of proteins, enzymes, etc. P.B.S.

Absorption Spectra of Dyes. IX— Changes of Absorption Curves caused by Exposure of Aqueous Solutions of Dyes to Light (IV p. 124)
Fastness to Light of Triphenylmethane Basic Dyes (IV p. 124)
Microscopic, Microchemical, and Physical Examination of the External and Internal Structure of Wool. II— Experimental Investigation of the Bilateral Structure and its Relation to Fibre Variations and Damage (VI p. 134)
Ion-exchange Celluloses for Chromatographic Separations (XI p. 143)
Graphic Test for Linear Fit to Kubelka–Munk Analysis (XV p. 148)

XV— MISCELLANEOUS

Theory of Colour Vision
M. Boynton *J. Opt. Soc. Amer.*, **50** (Oct 1960) 929–944

An attempt is made to improve on the Hurvich–Jameson theory of colour vision. Defects in this theory are listed and the facts of colour vision which are explained by the proposed modified theory are detailed. The theory is a quantitative, tristimulus, five-receptor, opponent-colours description of the human colour-vision process. It is assumed that there are three photopic visual pigments, the characteristics of which are described. Evidence is given in support of the choice of pigments whose absorption spectra, for low concentrations, are linear transformations of the C.I.E. tristimulus values for an equal-energy spectrum. The theory assumes that there are five types of foveal cone receptors, *R*, *Y*, *G*, *B*, and *W*, brightness depending upon the activity of a single type of cone *W*. Equations representing the distribution of the three pigments in each cone type are given, and opponent-colours sensitivity functions $(R-G)$, $(Y-B)$ are related to the C.I.E. distribution coefficients. Luminance refers to a weighted measure of stimulus energy which takes into account the spectral sensitivity of the eye to brightness, and a new term, chrominance, is introduced. This refers to a weighted measure of stimulus energy which takes into account the spectral sensitivity of the eye to colour. The generation and transmission of opponent-colours responses is discussed in terms of interacting potentials at the retinal level, which produce a d.c. potential characteristic of the stimulus. The recoding of the $(R-G)$ and $(Y-B)$ information to give a physiological response at the cortical level of red, green, yellow, and blue, and separately the *W* response, is discussed. Saturation is expressed as the ratio of chromaticness to brightness (subjective) and is related to chrominance and luminance in the form of a power ratio for each of the four chromatic responses. Brightness is taken as a power factor of luminance. In the process of these manipulations five constants appear which are assumed in the first place to be equal and of value unity. On this assumption equations for saturation and hue value are calculated in terms of chromaticity coordinates. A unit-*k* saturation–hue diagram is plotted, showing part of the spectrum locus and some of the Munsell colours at Value 5. For low saturations the diagram provides a reasonably uniform colour space but is distorted at higher saturations. A better colour space could be obtained by choice of more suitable values for the constants previously equated to unity. The theory accounts for the main features of colour blindness and there is some discussion of protanopia, deuteranopia, and tritanopia, in addition to the Bezold–Brücke effect and deviations from Abney's law.
E.Coates

Theory on the Receptor Mechanism in Colour Vision
C. Schroeder *J. Opt. Soc. Amer.*, **50** (Oct 1960) 945–949

In the theory presented, a model is explored in which the different colour responses are caused by different positions within a cone of identical photochemical receptors, and is concerned only with the first step in the total process of colour perception. The basis of the proposed mechanism is the interference of light waves which enter the outer cylindrical segment of a cone directly, with those which are reflected into this outer cylinder from the cone-shaped junction between outer and inner segments. This gives rise to standing waves along the outer segment. The maxima and minima of the waves will be in different positions according to the wavelength of light. Three photosensitive detectors are considered to be at different positions along the length of the segment which contains one photosensitive pigment (rhodopsin). A colour-mixture diagram is derived which approximates to the C.I.E. diagram, and reasons for several deviations from normal vision are suggested. E.Coates

Computer for the Conversion of Tristimulus Values to Trichromatic Coefficients
W. Preston *J. Opt. Soc. Amer.*, **50** (Nov 1960) 1117–1118

A computer is described which converts tristimulus data (X, Y, Z) to chromaticity coordinates (x, y). For some time a tristimulus integrator has been available for the Hardy spectrophotometer. This integrator automatically computes tristimulus figures as the reflectance curve of a sample is recorded. The present computer is designed to remove the last traces of effort needed to derive chromaticity coordinates, although there is little improvement in speed over manual methods. E.Coates

Colour Appearance Specification with Adaptation to Daylight and Tungsten Illumination
R. W. Burnham and R. J. Malsch
J. Opt. Soc. Amer., **50** (Nov 1960) 1071–1074

The Glasser modification of an Adams-type colour space has been evaluated for its capacity to take into account adaptation effects as they affect colour appearance. In an ideal colour-appearance space, where adaptation and

other effects are taken into account, matching colours under different adaptations would plot in the same location. Specifications for a large sample of the Munsell renotation colours reported by Newhall for source *C* were transformed to specifications for source *A* by use of the Burnham–Evans–Newhall equations. Both sets of specifications were then transformed to the Glasser cube-root space (which agrees well with the Adams space). Deviations from coincidence of matching colours under the two adaptations were determined. Departures from coincidence were large and complex, so that attempts to derive analytical expressions to bring about coincidence met with failure. The results show that the Adams-type colour space should be used with caution and typically only for colours viewed under source *C*, as a means of specifying colour appearance. E.COATES

Colorimetry and its Application in the Knitting Industry
Fr. Braun
Bull. Centre Recherches Bonneterie, No. 11 (1960) 17–30

An account is given of the fundamental principles of colorimetry. The basis of the C.I.E. system of colour specification is outlined, and spectrophotometric, tristimulus colorimetric, and spectrocolorimetric methods are described. Consideration is given to colour tolerance and the use of colorimetry in the study of colorant type and purity in connection with depth of shade. The possibility of matching colour patterns by the use of the Kubelka–Munk reflectance–dye concentration relation is mentioned and objections to the method are raised. A method for colour matching based on colorimetric measurement is discussed. Some eight basic colorants are used (from red to violet) and a colour gamut is constructed for a given depth of colour. The specification of these colours separately and in binary mixtures together with proportions of grey (red–yellow–blue) are determined and plotted for each depth of colour. The specifications for a sample to be matched are then considered in relation to these gamuts and the colour recipe is calculated. In the majority of cases an exact match is obtained directly and in other cases by approximation. E.COATES

Graphic Test for Linear Fit to Kubelka–Munk Analysis
R. S. Teague *J. Opt. Soc. Amer.*, **50** (Oct 1960) 1027–1028

Plots of log (K/S), corresponding to reflectance, against log[$f_b/(1-f_b)$], where f_b is the proportion of a non-scattering black component of a mixture containing a non-absorbing white, are shown. Straight parallel lines represent theoretical values conforming to the Kubelka–Munk analysis at four different ratios of K_b/S_w. Curves represent the theoretical values for a purely additive mixture and for a textile mixture used by Stearns, an empirical formulation. E.COATES

Economical Dyehouse Management
H. R. Bush *Dyer*, **124** (16 Dec 1960) 917–920

Discussion of improvements at small capital expense. Many commission dyehouses are old, with haphazard lay-out which disturbs flow. Examples from loose-stock dyehouses: uneven floors, unused machines, heavy manual transport— a string diagram would show basis for replanning. Time is saved by improved loading, unloading, and stuffing of machines. Even if general maintenance is good, electrical control is less so, e.g. faulty instruments. Colour stores, redyes, sampling, filing, and grey records are discussed. R.A.P.

Dynamic Programming in Chemical Technology— Optimisation of Yields in Chemical Reaction
W. F. Grütter and B. H. Messikommer
Helv. Chim. Acta, **63** (Nov 1960) 2182–2186

Bellman's theory (*Dynamic Programming*, Princeton University Press, 1957), developed in the field of operational research, is now explained by means of an example: the optimisation of a cascade of continuously stirred tank reactors with respect to yield, achieved by optimal distribution of the total residence time between the vessels. H.H.H.

Rôle of Chemical Research in the University and in Industry
E. Rideal *Chem. & Ind.*, No. 49 (3 Dec 1960) 1488–1491

General discussion with particular reference to the relation between technological and purely scientific research, and to the relative advantages and disadvantages of central organisation of research in totalitarian countries and the looser structures existing in the democracies. There is a tendency, particularly in the U.S.A., for organisation in the latter nations to approach that in the former. The essential nature of research is discussed. J.W.D.

Education in the Factory: Development of Personality the Final Target
A. M. Friedrich
Melliand Textilber., **41** (Oct 1960) 1292–1295

The work of a man in a factory can be judged from three standpoints: (*a*) the actual amount of work done, (*b*) whether done economically, i.e. with the minimum consumption of materials, tools, and energy, and without undue strain on machinery, and (*c*) whether the worker is conscious of his social duties and is prepared to assist his coworkers by giving actual help, by sharing his professional experiences, and whether his personality is such as to have a good influence on other workers. The great importance of the last point is stressed. W.M.

PATENTS

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U.S. Secretary of the Army *USP* 2,926,061 (23 Apr 1957)

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Moth- and Rot-proofing of Feathers
U.S. Secretary of the Army *USP* 2,925,315 (23 Apr 1957)

Treatment with aqueous acrolein at 35–46°C increases the filling power and resiliency of land-fowl feathers and also makes them resistant to moth and microbiological attack. C.O.C.

Improving the Wettability of Coloured Anodised Aluminium
Midwest Manufacturing Co. *USP* 2,924,539 (26 Oct 1956)

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USP 2,927,045 (17 Aug 1956)

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Stains for Cellulose, Paper, Wood, etc. (IV p. 130)
Philosophy of Coatings (V p. 131)
Sensitisation of Photoconductivity in Glass by Dyes (XIV p. 145)

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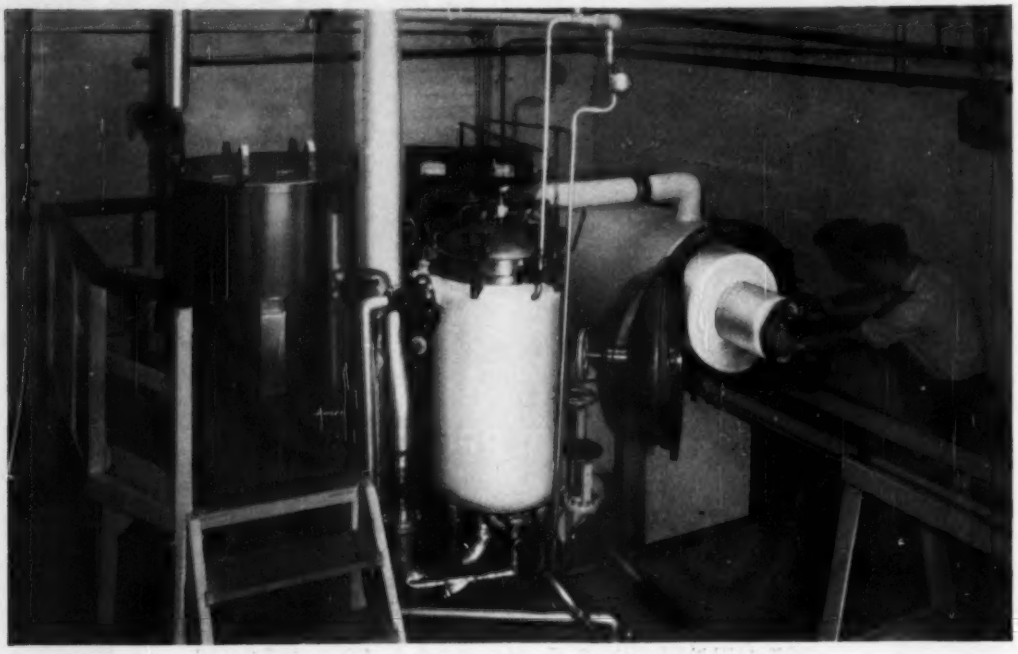

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continued on page xxxvii

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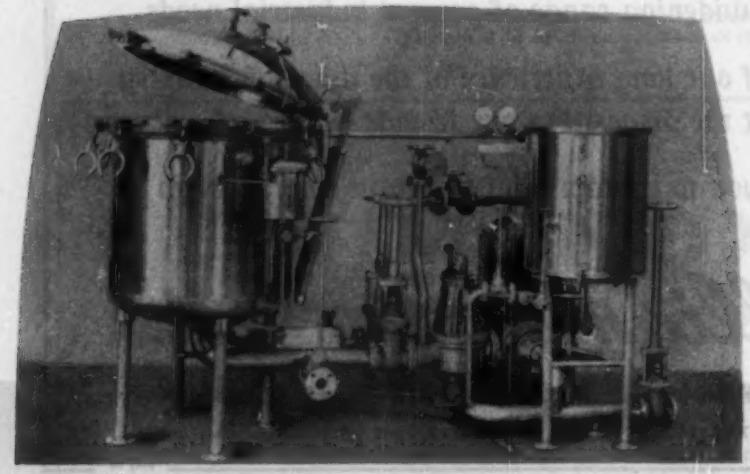

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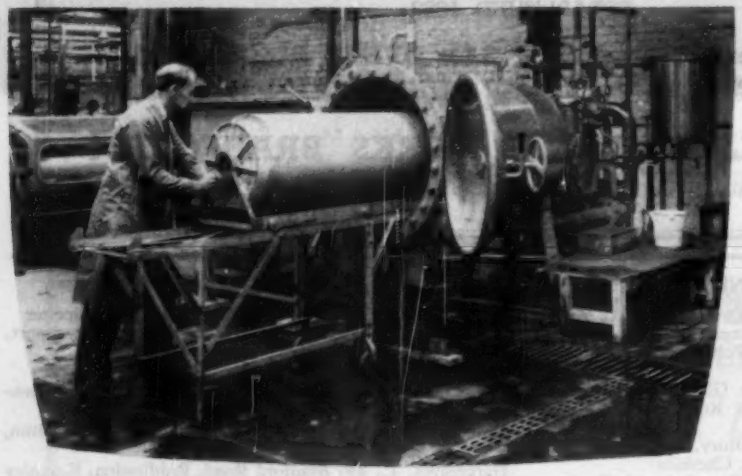

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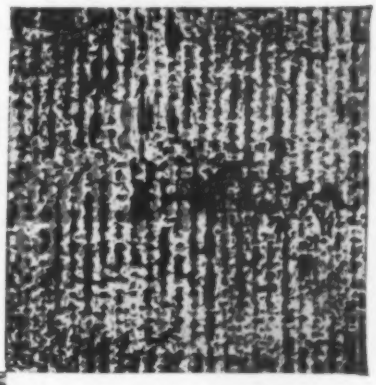

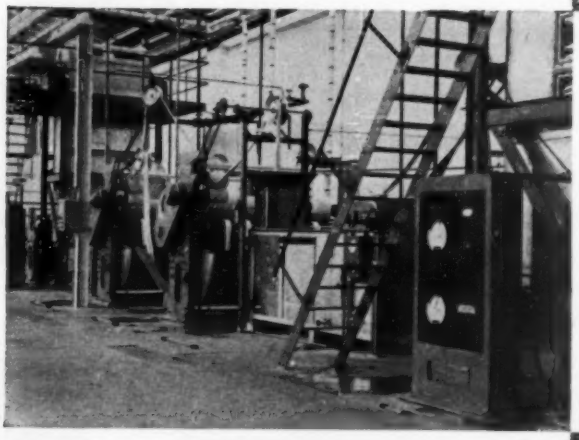

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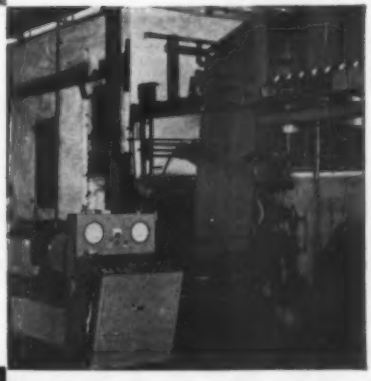

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ENTERED AT THE NEW YORK N.Y. U.S.A. POST OFFICE AS SECOND CLASS MATTER

REGISTERED FOR TRANSMISSION BY MAGAZINE POST TO CANADA